



## ORIGINAL ARTICLE

# Ultrasound as pre-treatment for microwave drying of *Myrtus communis* fruits: Influence on phenolic compounds and antioxidant activity

Nadia Bouaoudia-Madi <sup>1,2\*</sup>, Sofiane Dairi <sup>1,3</sup>, Omar Aoun <sup>1,2</sup>, Nabil Kadri <sup>2,4</sup>, Khodir Madani <sup>2</sup>, Lila Boulekbache-Makhlouf <sup>2</sup>

<sup>1</sup> Mouhamed Boudiaf University of Msila, Faculty of Sciences, Department of Microbiology and Biochemistry, Algeria. [nadia.madi@univ-bejaia.dz](mailto:nadia.madi@univ-bejaia.dz) / [bouaoudia.nadia@gmail.com](mailto:bouaoudia.nadia@gmail.com)

<sup>2</sup> University of Bejaia, Faculty of Sciences and Natural life, Laboratory of Biomathematics, Biophysics, Biochemistry, et Scientométrie (L3BS), 06000 Bejaia

<sup>3</sup> University of Jijel, Faculty of Sciences and Natural life, Department of Applied Microbiology and Food Sciences, 18000 Jijel, Algeria. [sofianedairi@yahoo.fr](mailto:sofianedairi@yahoo.fr)

<sup>4</sup> University of Bouira, Faculty of Sciences and Natural life, earth Sciences, Department of Biology, Bouira, Algérie. [Kadri.montp2@gmail.com](mailto:Kadri.montp2@gmail.com)

## Abstract

**Background:** Drying constitutes the most common method of food preservation that may degrade nutrients compounds in fruits due to high temperatures and long drying times. *Myrtus communis* is one of the important aromatic and medicinal species, owing to these reasons, the development of new methods of drying is essential for the preservation and valorization of myrtle fruits. **Aims:** The aim of the present study was to investigate the effect of ultrasound as a pre-treatment (USP) at 10 min to 90 min in microwave-drying (MD) on the dehydration of myrtle *Myrtus communis* fruits, on phytochemical content, and on antioxidant activity. **Methods:** ultrasound drying as pretreatment in microwave drying, extraction yield efficiency and antioxidant activity were measured using radical scavenging assay (DPPH•) and reducing power in addition the PCA analysis was investigated to detect the relationships between variables. **Results:** The ultrasound pretreatment reduced notably the microwave drying time. A pretreatment of 90 min provided the most rapid drying kinetics (6 min and 5.5 min for 500 w and 700 w respectively) compared to the microwave drying alone (18 min and 11 min for 500 w and 700 w respectively). A higher phytochemical content;  $219.90 \pm 0.69$  mg GAE/g for total phenol content (TPC) was obtained compared to those from MD and conventional drying (CD);  $193.79 \pm 0.99$  mg GAE/g and  $148.16 \pm 0.95$  mg GAE/g for TPC respectively. Indeed, the antioxidant activity tests revealed that ultrasound pretreatment is one of the most efficient methods to preserve the quality and the hydrogen and/or electron-donating ability of antioxidants for neutralizing DPPH radicals (98.63 %) test and reducing ferric ions to ferrous ones. Effectively, the results of PCA analysis show a higher positive correlation between antioxidant activity and flavonoids, anthocyanins, and tannins contents. **Conclusions:** Ultrasound pretreatment is expected to be a potential alternative to preserve fruit quality during microwave drying because it can reduce drying time at ambient temperatures while preserving natural heat-sensitive nutritive components, flavor, and color.

## Article information

**\*Corresponding author:** Nadia BouaoudiaMadi, Mouhamed Boudiaf University de Msila, Faculty of Sciences, Department of Microbiology and Biochemistry, Laboratory of Biomathematics, Biophysics, Biochemistry, et Scientométrie (L3BS), Email: [nadia.madi@univ-bejaia.dz](mailto:nadia.madi@univ-bejaia.dz) Tel. +213 (540 382 506).

**Received:** April 09, 2022

**Revised:** September 19, 2022

**Accepted:** September 26, 2022

**Published:** October 15, 2022

### Article edited by:

- Pr. Meghit B. Khaled

### Article reviewed by:

- Pr. Farid Dahmoune

- Dr. Amine Belbahi

- Dr. Midhat Nabil Bin Ahmad Salimi

### Keywords:

Ultrasound, *Myrtus communis* L, microwave, drying, pretreatment, antioxidant activity.

## 1 Introduction

Among the medicinal plants, *Myrtus communis* is one of the important aromatic and medicinal species from the Myrtaceae family <sup>1</sup>. This plant is also used in folk medicine because of its astringent and balsamic properties. The fruits are very rich in anthocyanin and are used as a condiment to substitute pepper. Before plan preservation, drying is the most common method of food preservation and is used to reduce post-harvest loss and to produce several dried fruits, which can be directly consumed or used in processed foodstuffs. Conventional air-drying is energy and cost intensive because it is a simultaneous heat and mass transfer process which can result in serious damage to flavor, color,

rehydration capacity and nutrients of the treated material as well as long low energy efficiency <sup>2</sup>. Therefore, development of new methods of drying for such perishable fruits (Myrtle) is essential for food preservation, which can economize time and energy and minimize quality degradation.

Since, the microwave drying has gained popularity as an alternative drying method to overcome above problems for a wide variety of food products <sup>3</sup>. However, microwave drying caused an excessive temperature in the food products and thus leads scorching and production of off-flavors especially during final stages of drying <sup>4</sup>. Hence, it is necessary to combine microwave

drying with a pretreatment to minimize product quality degradation.

In recent years, ultrasound has been implemented as an alternative pretreatment method for drying, and the results have shown that this pretreatment can significantly reduce the overall processing time<sup>5</sup> and<sup>6</sup> which can attribute to the following factors: increase in the mass transfer rate, loss of cellular adhesion, rupture of the cell walls and formation of large channels<sup>7</sup>. Therefore, the aim of this study was to evaluate the effect of ultrasound pretreatments on myrtle fruits drying kinetics. The influence of pretreatment on water loss, TPC, and their antioxidant activity were analyzed. The comparison between the microwave drying assisted by ultrasound pretreatment, microwave and conventional drying was also investigated.

## 2 Material and Methods

### 2.1 Preparation of samples

*Myrtus communis* fruits were collected at optimal maturity (January), from Addekar municipality (Bejaia, North-east of Algeria). Fruits were isolated manually from the aerial parts and washed with a tap and distilled water to remove any adhering soil and dust. Finally, fruits were blotted with filter paper and pretreated by ultrasound.

### 2.2 Ultrasound pretreatment microwave drying (USP-MD)

The samples (fruits) were immersed in distilled water, covered with the metal net to avoid flow out of the samples and placed in an ultrasonic bath (Ctra.NII:585 Abrera (Barcelona) Spain.). The pretreatment was carried out at room temperature (25 °C). The ratio of raw material to water was set at 1:4 (w/v), as recommended by Fernandes *et al.*<sup>8</sup>. The ultrasound frequency was set at 25 kHz and the ultrasound energy was applied for 10, 20, 30, 45 and 90 min respectively. The plant material was blotted with filter paper. Before and after ultrasound treatment, the mass of the samples, dry matter content and water temperature were measured. The water loss of the samples was measured after pretreatment with ultrasounds. After ultrasound pretreatment, the fruits were dried in microwave; domestic digital microwave oven with the technical feature of 230 V, 50Hz and 2450 W. The size of the heating cavity is 37.5 cm (L) x 22.5 cm (W) x 38.6 cm (D) by two power (500 W and 700 W). The microwave oven was operated by a control terminal, which could control microwave power level and emission time. After drying, fruit samples were peeled manually, and seeds were recovered. The pericarps were ground with an electrical grinder (IKA model A11 Basic, staufen, Germany). The obtained powder was passed through standard 125 µm size and only the fraction with particle size < 125 µm was utilized. The powder was stored in airtight bags until use.

### 2.3 Ultrasound assisted extraction (UAE)

Extraction of phenolic compounds, using ultrasound, was proposed to improve the efficiency and/or speed of this step. An ultrasonic apparatus (Vibra cell, VCX 75115 PB, SERIAL No.

2012010971MODEL CV 334, SONICS, Newtown, Connecticut, USA) was used for UAE with working frequency fixed at 20 kHz. For the extraction, one gram of the powder was placed in a 250 mL amber glass bottle. The suspension was exposed to acoustic waves with a concentration of 28 mL of ethanol at 70 %, irradiation time (7 min 30 s), and amplitude at 30%<sup>9</sup>. The temperature (27 ± 2 °C) was controlled continuously by circulating external cold water and checking the temperature using a T-type thermocouple. After the extraction, the solution was filtered through filter paper.

## 2.4 Analytical determinations

### 2.4.1 Total phenolic content (TPC)

The determination of total phenolic compounds in the extracts was done according to the method of Georgé *et al.*<sup>10</sup>. A volume of 500 µL of diluted pericarps extract was added to 2.5 mL of 10-fold diluted Folin–Ciocalteu reagent. The solution was mixed and incubated at room temperature for 2 min. Then, 2 mL of 7.5 % sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) (m/v) were added. After incubation at 50° C for 15 min, the absorbance of the sample was measured at 760 nm against a blank by using a UV–VIS Spectrophotometer (SpectroScan 50, Nkesia, Cyprus). The assay was performed in triplicate. For quantification, a calibration curve was generated with the standard solution of gallic acid (R<sup>2</sup>= 0.998) and the results were expressed as mg of gallic acid equivalent (GAE) per gram of powder on dry weight (DW) basis (mg GAE g<sup>-1</sup> DW).

### 2.4.2 Total flavonoid content

The total flavonoid content was estimated according to the aluminum chloride method of Quettier-Deleu *et al.*,<sup>11</sup> based on the formation of a complex flavonoid-aluminum<sup>12</sup>. 1 mL of pericarps extracts was mixed with 1 mL of 2 % AlCl<sub>3</sub>. After 15 min of incubation in the dark, the absorbance of the mixture was determined at 430 nm. Each analysis was carried out in triplicate. The total flavonoid content was calculated from a calibration curve made with rutin as standard and expressed as milligrams of rutin equivalent per gram of powder on dry weight (DW) basis (mg RE g<sup>-1</sup> DW).

### 2.4.3 Total monomeric anthocyanin content

Total monomeric anthocyanin content was determined by the pH-differential method<sup>13</sup>, based on the structural change of the anthocyanin chromospheres between pH 1.0 and 4.5. The absorbance was measured at 520 nm and at 700 nm in potassium chloride (KCl) at pH 1.0 (0.025 M) buffer and sodium acetate (CH<sub>3</sub>CO<sub>2</sub>Na) at pH 4.5 (0.5 M). The concentration of anthocyanin was obtained using the following equation (Eq. 1). Results are expressed on a cyanidin-3- glycoside basis.

$$\text{Anthocyanin pigment (cyanidin-3-glucoside equivalents, mg/g DW)} = \frac{A \times \text{MW} \times \text{FD} \times 10^3}{\epsilon \times L} \quad (1)$$

Where:

$$A = (A_{520\text{nm}} - A_{700\text{nm}})_{\text{pH}1.0} - (A_{520\text{nm}} - A_{700\text{nm}})_{\text{pH}4.5}$$

MW is the molecular weight (449.2 g/mol) for cyanidin-3-glucoside (cyd-3-glu).

DF is the dilution factor; L is the path length in cm and  $\epsilon$  is the molar extinction coefficient 26 900, in for cyd-3-glu ( $L \times \text{mol}^{-1} \times \text{cm}^{-1}$ ); and  $10^{-3}$  is the factor for conversion from g to mg.

#### 2.4.4 Total condensed tannin content

Total tannin content was determined by the HCL–Vanillin procedure according to Ba *et al.* <sup>14</sup>. 1 mL of the extract was mixed with 5 mL of reagent (HCL + Vanillin). The mixture was put in the dark for 20 minutes. The absorbance versus prepared blank was read at 500 nm. All analyses were performed in triplicate. Total tannins are expressed as mg catechin equivalents per gram (mg CE/g) through the calibration curve with catechin as standard.

### 2.5 Antioxidant activity

#### 2.5.1 DPPH radical scavenging assay

The free radical scavenging activity (RSA) was measured, following the method of Dudonne *et al.* <sup>15</sup>. A DPPH<sup>•</sup> solution in absolute methanol (60  $\mu\text{M}$ ) was prepared, and 3 mL of this solution were mixed with 1 mL of different diluted extracts. The samples were incubated for 20 min at 37°C in the dark, then, the absorbance at 515 nm was measured. The  $\alpha$ -tocopherol served as a positive control. All the tests were performed in triplicate and the inhibition rate was calculated according to the following equation (Eq. 2).

$$\% \text{ Scavenging} = \frac{(A_{\text{control}} - A_{\text{extract}})}{A_{\text{control}}} \times 100 \quad (2)$$

Where,  $A_{\text{control}}$  is the absorbance of DPPH solution at time 0 min and  $A_{\text{sample}}$  is the absorbance of DPPH solution at time of 20 min.

#### 2.5.2 Iron reducing power

According to the method described by Pan *et al.* <sup>16</sup>, 1 mL of desired dilution with distilled water of pericarps extracts was mixed with 2.5 mL of sodium phosphate buffer (0.2 M, pH 6.6) and 2.5 mL of 1 % potassium ferricyanide ( $\text{K}_3\text{Fe}(\text{CN})_6$ ). The mixture was incubated in a water bath at 50 °C for 20 min. Then, 2.5 mL of 10 % trichloroacetic acid were added. Finally, 1 mL of the obtained solution was added to 5 mL of distilled water and 1 mL of 0.1 % ferric chloride ( $\text{FeCl}_3$ ). The intensity of the blue green color was measured at 700 nm. The higher absorbance corresponds to the higher antioxidant activity. Tests were carried out in triplicate.

### 2.6 Statistical analysis

The analysis of variance (ANOVA) was performed using XLSTAT release 10 (Addinsoft, Paris, France), Tukey's multiple range test (HSD) was used to compare between TPC content and antioxidant activity as affected by microwave-assisted by ultrasound (USP-MD), microwave (MAE) and conventional drying methods (CD). Principal component analysis (PCA) was performed to highlight the relationships between variables. PCA

was applied to the phenolic compounds; flavonoid, anthocyanins, tannin, content, and antioxidant activity of myrtle extract obtained with different drying methods (ME CE and USP-MD), and two factors were selected justifying 100 % of the total variance.

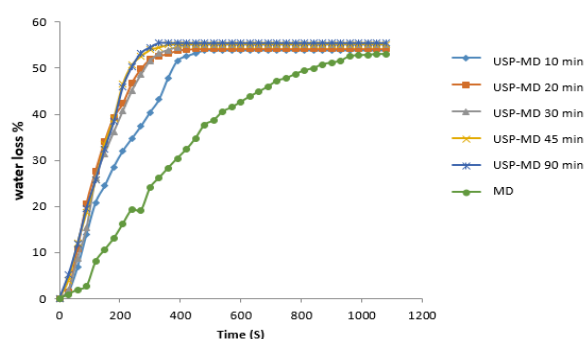
## 3 Results

### 3.1 Ultrasound pretreatment-assisted microwave drying (USP-MD)

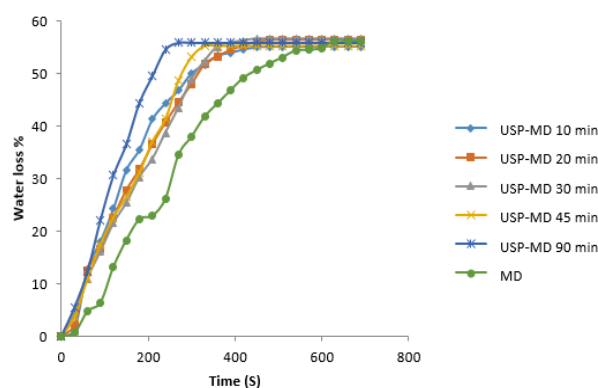
Drying kinetics were studied until a final water loss of  $56 \pm 0.005$  %. In the present study, the pretreatment with ultrasound for 10, 20 and 30 min had a positive effect on water loss of myrtle fruit tissue compared to the MD treatment alone (500 and 700 W) (Figures 1 and 2), especially in the first 30 min of the treatment.

### 3.2 Analytical determination

#### 3.2.1 Total phenolic content (TPC)



**Figure 1:** Influence of ultrasonic pretreatment time on the microwave dehydration kinetic process at 500 W of myrtle pericarp



**Figure 2:** Influence of ultrasonic pretreatment time on the microwave dehydration kinetic process at 700 W of myrtle pericarp

The phenolic composition of dried myrtle fruits obtained with USP-MD at 500 W, and 700 W is summarized in Table 1. The

major significant differences were observed in the TPC content between the different ultrasonic pretreatment times; with an optimal content about  $219.90 \pm 0.69$  mg GAE /g at 500 W and  $204.43 \pm 0.43$  mg GAE /g at 700 W.

### 3.2.2 Total flavonoid content

The total flavonoid content showed a significant difference ( $p < 0.05$ ) between all samples Table 1. The highest amount was attributed to sample pretreated for 90 min ( $5.75 \pm 0.01$  mg RE / g), and the lowest content was found with the sample pre-treated for 10 min ( $3.69 \pm 0.05$  mg RE/g), and this is in agreement with TPC content.

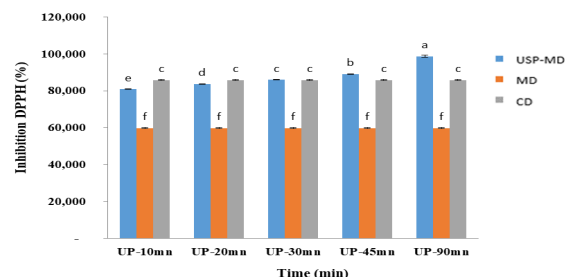
### 3.2.3 Total Anthocyanins content

As summarized in Table 1, the results showed that the highest content was observed in samples pretreated by ultrasound at 90 min with microwave drying at 500 W ( $11.30 \pm 0.02$  mg cy-3-glu E/g), and the lowest yield was observed by 10 min ( $6.46 \pm 0.06$  mg cy-3-glu E /g).

### 3.2.4 Total condensed tannins content

The results in Table 1 show that, condensed tannin content, in the samples, obtained with microwave drying at 500W, after ultrasounds pretreatment, was the highest with a significant difference ( $p < 0.05$ ) between all samples, compared to those obtained for 700 W.

The results of radical DPPH scavenging activity of myrtle extract obtained with different drying methods was illustrate in Figure 3. The extracts obtained by microwave drying at 500 W pretreated by ultrasound show that the inhibition effect of DPPH radical antioxidant was most important.



**Figure 3:** Radical DPPH scavenging activity of myrtle extract obtained with different drying methods

#### • Iron reducing power

The results in Figure 4 show that the highest absorbance was observed for fruits pretreated at 90 min at 500 W, and the lowest was attributed to those pretreated at 10 min. The same relationship between antioxidant activity and phenolic content was observed. These observations were confirmed by Hrenovic *et al.* <sup>17</sup>.

**Table 1:** Phenolic composition of dried myrtle fruits obtained with USP-MD at 500 W and 700 W

|              | Method conditions | Recovery of TPC (mg GAE / g) | Recovery of flavonoid (mg RE / g) | Recovery of anthocyanins (CE mg / g) | Recovery of tannin (mg QE / g) |
|--------------|-------------------|------------------------------|-----------------------------------|--------------------------------------|--------------------------------|
| <b>500 W</b> | USP-10 min        | $115.78 \pm 0.26^i$          | $3.69 \pm 0.05^c$                 | $6.46 \pm 0.06^e$                    | $111 \pm 0.54^h$               |
|              | USP-20 min        | $140.25 \pm 0.77^b$          | $3.81 \pm 0.05^d$                 | $6.86 \pm 0.1^e$                     | $119.83 \pm 0.8^g$             |
|              | USP-30 min        | $160.34 \pm 0.22^c$          | $4.10 \pm 0.01^c$                 | $7.99 \pm 0.40^d$                    | $136.38 \pm 0.28^f$            |
|              | USP-45 min        | $173.11 \pm 0.18^b$          | $4.70 \pm 0.09^b$                 | $10.51 \pm 0.01^b$                   | $158.6 \pm 0.46^c$             |
|              | USP-90 min        | $219.90 \pm 0.69^a$          | $5.75 \pm 0.01^a$                 | $11.30 \pm 0.02^a$                   | $200.56 \pm 0.76^a$            |
| <b>700 W</b> | USP-10 min        | $108.84 \pm 0.43^j$          | $2.49 \pm 0.01^h$                 | $5.79 \pm 0.06^f$                    | $85.61 \pm 0.72^i$             |
|              | USP-20 min        | $130.82 \pm 0.03^h$          | $2.80 \pm 0.04^g$                 | $6.46 \pm 0.47^e$                    | $110.06 \pm 1.6^h$             |
|              | USP-30 min        | $154.17 \pm 0.36^f$          | $3.17 \pm 0.03^f$                 | $6.98 \pm 0.17^e$                    | $153.15 \pm 0.9^d$             |
|              | USP-45 min        | $167.10 \pm 0.24^c$          | $3.85 \pm 0.005^d$                | $7.95 \pm 0.02^d$                    | $141.86 \pm 0.16^e$            |
|              | USP-90 min        | $204.43 \pm 0.43^b$          | $4.75 \pm 0.04^b$                 | $9.47 \pm 0.02^c$                    | $192.23 \pm 0.83^b$            |

Assays performed in triplicate ( $n = 3$ ) and data expressed as mean  $\pm$  standard deviation. Means with different letters within each column are significantly different  $p < 0.05$ .

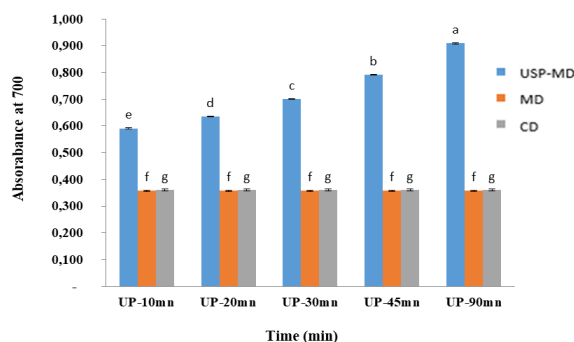
### 3.2.5 Antioxidant activity

#### • DPPH radical scavenging assay

## 4 Discussion

#### 4.1 Ultrasound pretreatment-assisted microwave drying (USP-MD)

The use of ultrasound in the food industry is new; only a few studies have addressed the use of ultrasound pre-treatment in drying processes. Tarleton <sup>18</sup>; Mason *et al.* <sup>19</sup>; Tarleton & Wakeman <sup>20</sup>; Gallego-Juárez *et al.* <sup>21</sup>; Fuente-Blanco *et al.* <sup>22</sup>;



**Figure 4:** Reducing power of myrtle extract obtained with different drying methods

Zheng & Sun <sup>23</sup>; Fernandes & Rodrigues <sup>24</sup>, Rodríguez-Fernández <sup>25</sup>, most of them dealing with ultrasound assisted osmotic dehydration and ultrasound assisted spray-drying.

Ultrasound pretreatment applied prior to microwave drying (MD), improves the mass transfer phenomena and the duration of drying time of many fruits <sup>8, 26</sup>. The USP-MD affects the drying time that shows (6 min, 5 min 30 s at 500 W and 700 W respectively) compared with microwave drying (18 min at 500 W, 11mn at 700w). This could be due to the microscopic channels creation during ultrasound pretreatment, which may ease moisture removal and increase the diffusivity of the water <sup>8</sup>. The results illustrate that the ultrasonic pretreatment is interesting when the quantity of water in the fruit was very high such in our case.

The ultrasonic waves can cause a rapid series of compression and alternative expansions, in a manner <sup>27</sup> similar to a sponge when it is tightened and released on several occasions. The forces implied in this mechanical process can be much larger than those due to the surface tension, which holds moisture inside the capillaries of the fruit creating the microscopic channels, which can relieve the removal of moisture <sup>22</sup>. This result is in agreement with the observations of De la Fuente-Blanco <sup>22</sup>, who reported that increased water diffusivity, during the microwave drying process, reduced the time required for drying. In addition, the microwave drying with high temperature and long-time treatments can cause phenolic compounds degradation <sup>28</sup>. The advantage of ultrasound is that the process can be carried out at room temperature as no heating is required, reducing the potential of thermal degradation <sup>29</sup>.

#### 4.2 Analytical determination

##### • TPC

The highest level was found in the pretreated sample 90 min at 500 W (219.90 ± 0.69 mg GAE/g), however, the lowest was observed with a sample pretreated for 10 min (115.78 ± 0.26 mg GAE/g) which correspond to 47 % TPC reduction compared to 90 min result. These observations are in correlation with kinetic drying results, i.e., increase ultrasound pretreatment time, reduces MD time, and preserves TPC content from thermo-degradation. Microwave drying at 700 W showed that TPC content had the same tendency that 500 W one, but TPC yield was decreased by 7%. This lowest content could be caused by the thermal degradation of the phytochemicals at a higher microwave power <sup>30</sup>. Fernandes *et al.* <sup>30</sup> showed that after 180 minutes under osmotic dehydration, papayas can gain from 144.1% of sugar when the osmotic dehydration is carried out at 50°Brix and 50°C to 189.6% of sugar when the process is carried out at 70°Brix and 70°C. The results showed that ultrasonic treated papayas can have 13.8% less sugar than the dried fruit and at least 64.7% less sugar than the osmo-dehydrated fruit. As such, the ultrasonic treatment may be an interesting process to produce low sugar dried fruits.

##### • Total flavonoid

The content was higher in the sample pretreated for 90 min (5.75 ± 0.01 mg RE / g), which was explained by Oliveira *et al.* <sup>28</sup>, and Fernandes *et al.* <sup>30</sup> who studied dehydration of Malay apple using ultrasound as pretreatment explained that to reduce the initial moisture content of the fruit by 90%. The total processing time can be reduced by 233 min when Malay apples are subjected to ultrasound during 60 min that leading to better preservation of nutrients content. The result was also confirmed by Masoumeh Moghimi *et al.* <sup>31</sup> who demonstrated that in the ultrasound pretreatment, the power of the applied wave affected the oil extraction efficiency rather than irradiation time. With the application of ultrasound waves, due to the decomposition of cells' membrane and enhancements of holes numbers, the quantity of the extracted oil will increase.

##### • Total Anthocyanins content

The results can be explained by the reduction of time sonification in the ultrasonic bath, which can contribute to the preservation of nutritive compounds <sup>26</sup>. When Corrales *et al.* <sup>32</sup> extracted the available anthocyanins in grape waste with the use of ultrasounds, they demonstrated that 1 h after the extraction process, total phenolic compounds of the treated samples with the use of novel pretreatment approaches was 5% higher compared to control sample. However, the microwave drying at 700 W displayed an amount of 9.46 ± 0.02 mg cy-3-glu E/g; the lowest content of anthocyanin could be due to its degradation by the high temperatures and long-times irradiation in the microwave <sup>22</sup>.

##### • Total condensed tannin content

The results showed that the samples treated by microwave drying at 500 W, pre-sonicated at 90 min were the highest with a significant difference ( $p < 0.05$ ) (200.50 ± 0.76 mg EC/g).



Followed by sample pre-sonicated at 45 min with an amount of ( $158.6 \pm 0.46$  mg CE/g), while the lowest content ( $111 \pm 0.54$  mg CE/g) was attributed to the sample pre-sonicated for 10 min. This is because ultrasonic pretreatment minimizes degradation of bioactive compounds caused by the higher temperatures of microwave heating <sup>30</sup>. Furthermore, the condensed tannins content obtained with drying power of 700 W was  $192.23 \pm 0.84$  mg CE/g. This result may be due to the degradation of these compounds under the influence of microwaves irradiation <sup>8</sup>.

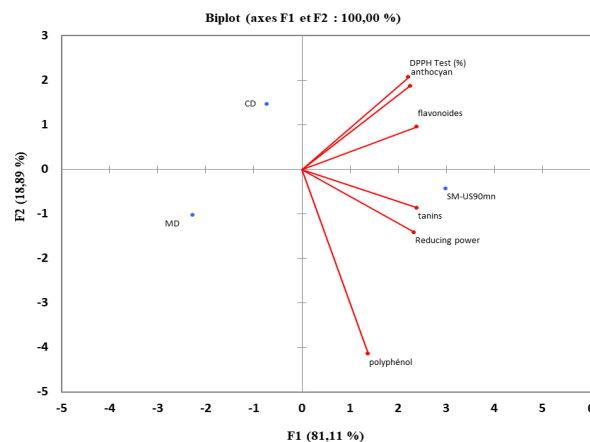
### 4.3 Antioxidant activity

The effect of antioxidants on DPPH radical scavenging was conceived to their hydrogen donating ability <sup>26</sup>. Concerning the extracts obtained by microwave drying at 500 W pretreated by ultrasound, Figure 3 shows that the most the inhibition effect of DPPH radical for sample pretreated for 90 min (98.63 %), followed by that pretreated for 45 min, and the lowest is recorded with that pretreated for 10 min (80.80 %). A positive correlation was recorded between DPPH scavenging activity and total phenolic compounds (flavonoids, tannins, and anthocyanins). These results were in agreement with those obtained by De la Fuente-Blanco *et al.* <sup>22</sup>, who demonstrated that high total polyphenol contents increase the antioxidant activity. Effectively, the sample drying with microwave at 700 W presented a low content of bioactive compounds comparison with those of 500 W, and thus, showing a lower antioxidant activity than myrtle dried at 500 W. Our results agree with those found by Yang *et al.* <sup>33</sup>. Identical outcomes were obtained when reducing power test providing the highest antioxidant activity for sample pretreated for 90 min with the same correlation between reducing power and phenolic compounds.

This result was confirmed by Rashid *et al.* <sup>34</sup> in drying of sweet potatoes using multi frequency ultrasound 1\*/-/ 01d (US) pretreatments (20, 40, and 60 kHz) at three different infrared (IR) drying temperatures (60, 70, and 80 °C). The antioxidant activity of the samples increased especially at 60 kHz and 80 °C, while US-IR treatments showed a positive effect on total phenolic compounds. The study provides evidence that infrared drying application in synergy with ultrasonic pretreatments can improve drying efficiency and food quality better than using each method isolated. The findings showed that moderate ultrasound frequency (40 kHz) at 60 °C improved phytochemical properties while antioxidant activities showed better preservation response at 80 °C with 60 kHz.

### 4.4 Comparison of USP-MD, MD and conventional drying methods

The selection of a drying method would mainly depend on the advantages and disadvantages of the processes, such as the drying time, extraction yield, complexity, production cost, environmental friendliness, and safety. Conventional air-drying is a simultaneous heat and mass transfer process, accompanied by phase change being a high-cost process. Usually, some form of pretreatment is used to reduce the initial water content or to modify the fruit tissue structure to reduce the total drying processing time <sup>35,36</sup>.



**Figure 5:** Principal component analysis of myrtle fruits drying methods

The conditions of different techniques and their results are summarized in Table 2. The results indicated that the highest TPC, total flavonoids, anthocyanins, and tannin content were found in extracts obtained by the USP-MD. That gave significantly higher values ( $p < 0.001$ ) at 500 W assisted by

**Table 2:** Phenolic composition of dried myrtle fruits obtained with different drying methods (USP-MD, MD, and CD)

| Drying methods       | Recovery of TPC (mg GAE/g) | Recovery of flavonoid (mg RE/g) | Recovery of anthocyanins (mg Cy 3 glu E/g) | Recovery of tannin (mg CE/g) |
|----------------------|----------------------------|---------------------------------|--|------------------------------|
| <b>USP-MD 90 min</b> | $219.90 \pm 0.69^a$        | $5.75 \pm 0.01^a$               | $11.30 \pm 0.02^a$                         | $200.56 \pm 0.76^a$          |
| <b>MD</b>            | $193.79 \pm 0.99^b$        | $4.41 \pm 0.10^c$               | $0.85 \pm 0.01^c$                          | $18.22 \pm .02^c$            |
| <b>CD</b>            | $148.16 \pm 0.95^c$        | $5.03 \pm 0.06^b$               | $7.43 \pm 0.20^b$                          | $41.38 \pm 0.5^b$            |

Assays are performed in triplicate ( $n = 3$ ) and data are expressed as mean  $\pm$  standard deviation. Means with different letters within each column are significantly different at  $p < 0.05$ .

ultrasound pretreatment of 90 min ( $p < 0.05$ ) compared with those obtained by microwave and conventional drying ( $p < 0.05$ ). Concerning the antioxidant activity, a comparison of different samples was presented in Figures 3 and 4. Using both assays (DPPH and Reducing power), the antioxidant activity was higher in pericarp extracts obtained by USP-MD compared to MD and CD ( $p < 0.05$ ). The efficiency of ultrasound pretreatment was to minimize the bioactive compound degradation caused by the higher temperatures of microwave heating <sup>37</sup>. The combination of microwave and ultrasound can be carried out at ambient temperature <sup>22</sup>, which has been proven by the works of Yang *et al.* <sup>33</sup>. The Fernandes *et al.*'s study <sup>26</sup> on the apples and the pineapples, showed that such a combination provides high speeds of water removal even at low temperatures,

thus leading to better maintenance of a natural aroma, color, and nutriment content.

#### 4.5 PCA analysis

In this study, PCA was applied to the phenolic compounds. Flavonoids, anthocyanins, tannin contents, and antioxidant activity of extracts obtained by different drying methods. Two factors were selected justifying 100 % of the total variance PC1 that explained 81.11 % of the total variance in the data set, whereas PC2 explained 18.89 %. The sample score plot for PC1 vs. PC2 is displayed in Figure 5. The position of each variable in this loading plot describes its relationship to the other variables. The figure shows the positive correlation between USP-MD and phenolic, flavonoids tannins, and anthocyanins compounds that are ported positively by PC1. However, CD and MD were ported negatively by PC1. Using the plots (Figure 5), it is possible to select the drying adequate method providing a higher antioxidant content. The best level was observed in the extract abstained by the USP-MD drying and ultrasounds assisted extraction explaining the higher correlation. In terms of antioxidant activity, DPPH and the reductive power test are positively correlated with polyphenol tannin compounds. The results of this study revealed the importance of comparing and exploring the variance of the phenolic composition with drying methods of myrtle fruits.

#### 5 Conclusion

Referring to the results of this investigation, pretreatment of myrtle fruits using ultrasound has led to accelerated water loss and improved the efficiency of microwave drying. The pretreatment time was also important in the drying process. In fact, among the pretreated myrtle fruits, the USP-90 min sample offered the fastest drying conditions. In addition, pretreated ultrasonic samples showed the best phenolic composition with a higher antioxidant activity than in the use of MD and CD processes. Thus, the utilization of ultrasound pretreatment may be the most appropriate alternative to preserve fruit quality during microwave drying because it can reduce drying time while preserving natural heat-sensitive nutritive components, flavor, and color.

**Source(s) of support:** No external support, grant or drug aid was received for this study.

**Acknowledgment:** The authors gratefully acknowledge the Algerian Ministry of Higher Education and Scientific Research for funding the study. The first author also wishes to thank the members of the research team of the laboratory: BBBS (University of Bejaia).

**Author Contribution:** B.N. designed the study, undertook the documentary research, carried out all the experiments and the acquisition, data analysis and article writing. D.S., A.O. and K.N. participated in the analysis of the data and in the editing of the article. M.K. and B.L. approved the final version before submission. All authors have read and accepted the published version of the manuscript.

No conflict of interest in this present study.

#### References

- [1] Aidi Wannes, W., Mhamdi, B., Sriti, J., Ben Jemia, M., Ouchikh, O., Hamdaoui, G., Kchouk, M. E., & Marzouk, B. (2010). Antioxidant activities of the essential oils and methanol extracts from Myrtle (*Myrtus communis* Var. *italica* L.) leaf, stem and flower. *Food and Chemical Toxicology*, 48 (5), 1362-1370. <https://doi.org/10.1016/j.fct.2010.03.002>
- [2] Özbek, B., & Dadali, G. (2007). Thin-layer drying characteristics and modelling of mint leaves undergoing microwave treatment. *Journal of Food Engineering*, 83 (4), 541-549. <https://doi.org/10.1016/j.jfoodeng.2007.04.004>
- [3] Bouraoui, M., Richard, P., & Durance, T. (1994). Microwave and convective drying of potato slices. *Journal of Food Process Engineering*, 17 (3), 353-363. <https://doi.org/10.1111/j.1745-4530.1994.tb00343.x>
- [4] Zhang, M., Tang, J., Mujumdar, A., & Wang, S. (2006). Trends in microwave-related drying of fruits and vegetables. *Trends in Food Science & Technology*, 17 (10), 524-534. <https://doi.org/10.1016/j.tifs.2006.04.011>
- [5] Jangam, S. V. (2011). An overview of recent developments and some R&D challenges related to drying of foods. *Drying Technology*, 29 (12), 1343-1357. <https://doi.org/10.1080/07373937.2011.594378>
- [6] Mothibe, K. J., Zhang, M., Nsor-atindana, J., & Wang, Y. (2011). Use of ultrasound Pretreatment in drying of fruits: Drying rates, quality attributes, and shelf life extension. *Drying Technology*, 29 (14), 1611-1621. <https://doi.org/10.1080/07373937.2011.602576>
- [7] García-Pérez, J. V., Ozuna, C., Ortuño, C., Cárcel, J. A., & Mulet, A. (2011). Modeling ultrasonically assisted convective drying of eggplant. *Drying Technology*, 29 (13), 1499-1509. <https://doi.org/10.1080/07373937.2011.576321>
- [8] Fernandes, F. A., Gallão, M. I., & Rodrigues, S. (2008). Effect of osmotic dehydration and ultrasound pre-treatment on cell structure: Melon dehydration. *LWT - Food Science and Technology*, 41 (4), 604-610. <https://doi.org/10.1016/j.lwt.2007.05.007>
- [9] Bouaoudia-Madi, N., Boulekbache-Makhlouf, L., Madani, K., Silva, A., Dairi, S., Oukhmanou-Bensidhoum, S., & Cardoso, S. M. (2019). Optimization of ultrasound-assisted extraction of polyphenols from *Myrtus communis* L. Pericarp. *Antioxidants*, 8 (7), 205. <https://doi.org/10.3390/antiox8070205>
- [10] Georgé, S., Brat, P., Alter, P., & Amiot, M. J. (2005). Rapid determination of polyphenols and vitamin C in plant-derived products. *Journal of Agricultural and Food Chemistry*, 53 (5), 1370-1373. <https://doi.org/10.1021/jf048396b>
- [11] Quettier-Deleu, C., Gressier, B., Vasseur, J., Dine, T., Brunet, C., Luyckx, M., Cazin, M., Cazin, J., Bailleul, F., & Trotin, F. (2000). Phenolic compounds and antioxidant activities of

- buckwheat (*Fagopyrum esculentum* Moench) hulls and flour. *Journal of Ethnopharmacology*, 72 (1-2), 35-42. [https://doi.org/10.1016/s0378-8741\(00\)00196-3](https://doi.org/10.1016/s0378-8741(00)00196-3)
- [12] Chang, C., Yang, M., Wen, H., & Chern, J. (2020). Estimation of total flavonoid content in propolis by two complementary colometric methods. *Journal of Food and Drug Analysis*, 10 (3). <https://doi.org/10.38212/2224-6614.2748>
- [13] Lee, J., Durst, R. W., Wrolstad, R. E., Eisele, T., Giusti, M. M., Hach, J., Hofsommer, H., Koswig, S., Krueger, D. A., Kupina, S., Martin, S. K., Martinsen, B. K., Miller, T. C., Paquette, F., Ryabkova, A., Skrede, G., Trenn, U., & Wightman, J. D. (2005). Determination of total monomeric anthocyanin pigment content of fruit juices, beverages, natural colorants, and wines by the pH differential method: Collaborative study. *Journal of AOAC INTERNATIONAL*, 88(5), 1269-1278. <https://doi.org/10.1093/jaoac/88.5.1269>
- [14] Ba, K., Tine, E., Destain, J., Cissé, N., & Thonart, P. (2010). Étude comparative des composés phénoliques, du pouvoir antioxydant de différentes variétés de sorgho sénégalais et des enzymes amylolytiques de leur malt. *Biotechnologie, Agronomie, Société et Environnement*, 14(1):131-139.
- [15] Dudonné, S., Vitrac, X., Coutière, P., Woillez, M., & Mérillon, J. M. (2009). Comparative study of antioxidant properties and total phenolic content of 30 plant extracts of industrial interest using DPPH, ABTS, FRAP, SOD, and ORAC assays. *Journal of Agricultural and Food Chemistry*, 57(5), 1768-1774. <https://doi.org/10.1021/jf803011r>
- [16] Pan, Y., He, C., Wang, H., Ji, X., Wang, K., & Liu, P. (2010). Antioxidant activity of microwave-assisted extract of *buddleia officinalis* and its major active component. *Food Chemistry*, 121(2), 497-502. <https://doi.org/10.1016/j.foodchem.2009.12.072>
- [17] Hrenovic, J., Milenkovic, J., Ivankovic, T., & Rajic, N. (2012). Antibacterial activity of heavy metal-loaded natural zeolite. *Journal of Hazardous Materials*, 201-202, 260-264. <https://doi.org/10.1016/j.jhazmat.2011.11.079>
- [18] Tarleton, E. (1992). The role of field-assisted techniques in solid/liquid separation. *Filtration & Separation*, 29(3), 246-238. [https://doi.org/10.1016/0015-1882\(92\)80255-h](https://doi.org/10.1016/0015-1882(92)80255-h)
- [19] Mason, T., Paniwnyk, L., & Lorimer, J. (1996). The uses of ultrasound in food technology. *Ultrasonics Sonochemistry*, 3 (3), S253-S260. [https://doi.org/10.1016/s1350-4177\(96\)00034-x](https://doi.org/10.1016/s1350-4177(96)00034-x)
- [20] Tarleton, E.S. & Wakeman, R.J. (1998). Ultrasonically assisted separation process. In: *Ultrasonics in Food Processing*; Povey, M.J.W.; Mason, T.J. Eds.; Blackie Academic and Professional, Glasgow, 193-218..
- [21] Gallego-Juarez, J. A., Rodriguez-Corral, G., Gálvez Moraleda, J., & Yang, T. S. (1999). A new high-intensity ultrasonic technology for food dehydration. *Drying Technology*, 17 (3), 597-608. <https://doi.org/10.1080/07373939908917555>
- [22] De la Fuente-Blanco, S., Riera-Franco de Sarabia, E., Acosta-Aparicio, V., Blanco-Blanco, A., & Gallego-Juárez, J. (2006). Food drying process by power ultrasound. *Ultrasonics*, 44, e523-e527. <https://doi.org/10.1016/j.ultras.2006.05.181>
- [23] Zheng, L., & Sun, D. (2006). Innovative applications of power ultrasound during food freezing processes - a review. *Trends in Food Science & Technology*, 17 (1), 16-23. <https://doi.org/10.1016/j.tifs.2005.08.010>
- [24] Fernandes, F. A., & Rodrigues, S. (2007). Ultrasound as pre-treatment for drying of fruits: Dehydration of banana. *Journal of Food Engineering*, 82 (2), 261-267. <https://doi.org/10.1016/j.jfoodeng.2007.02.032>
- [25] Rodríguez-Fernández, M., Balsa-Canto, E., Egea, J., & Banga, J. (2007). Identifiability and robust parameter estimation in food process modeling: Application to a drying model. *Journal of Food Engineering*, 83 (3), 374-383. <https://doi.org/10.1016/j.jfoodeng.2007.03.023>
- [26] Fernandes, F. A., Gallão, M. I., & Rodrigues, S. (2009). Effect of osmosis and ultrasound on pineapple cell tissue structure during dehydration. *Journal of Food Engineering*, 90 (2), 186-190. <https://doi.org/10.1016/j.jfoodeng.2008.06.021>
- [27] Rodrigues, S., & Fernandes, F. A. (2007). Use of ultrasound as Pretreatment for dehydration of melons. *Drying Technology*, 25 (10), 1791-1796. <https://doi.org/10.1080/07373930701595409>
- [28] Oliveira, F. I., Gallão, M. I., Rodrigues, S., & Fernandes, F. A. (2010). Dehydration of Malay Apple (*Syzygium malaccense* L.) using ultrasound as pre-treatment. *Food and Bioprocess Technology*, 4 (4), 610-615. <https://doi.org/10.1007/s11947-010-0351-3>
- [29] Povey MJ, Mason TJ: Ultrasound in food processing: Springer Science & Business Media; 1998.
- [30] Fernandes, F. A., Oliveira, F. I., & Rodrigues, S. (2007). Use of ultrasound for dehydration of papayas. *Food and Bioprocess Technology*, 1 (4), 339-345. <https://doi.org/10.1007/s11947-007-0019-9>
- [31] Moghimi, M., Farzaneh, V., & Bakhshabadi, H. (2018). The effect of ultrasound pretreatment on some selected physicochemical properties of Black cumin (*Nigella Sativa*). *Nutrire*, 43 (1). <https://doi.org/10.1186/s41110-018-0077-y>
- [32] Corrales, M., Toepfl, S., Butz, P., Knorr, D., & Tauscher, B. (2008). Extraction of anthocyanins from grape by-products assisted by ultrasonics, high hydrostatic pressure or pulsed electric fields: A comparison. *Innovative Food Science & Emerging Technologies*, 9 (1), 85-91. <https://doi.org/10.1016/j.ifset.2007.06.002>
- [33] Yang, Z., & Zhai, W. (2010). Optimization of microwave-assisted extraction of anthocyanins from purple corn (*Zea mays* L.) COB and identification with HPLC-MS. *Innovative Food Science & Emerging Technologies*, 11 (3), 470-476. <https://doi.org/10.1016/j.ifset.2010.03.003>
- [34] Rashid, M. T., Ma, H., Jatoti, M. A., Wali, A., El-Mesery, H. S., Ali, Z., & Sarpong, F. (2019). Effect of infrared drying with multifrequency ultrasound



- pretreatments on the stability of phytochemical properties, antioxidant potential, and textural quality of dried sweet potatoes. *Journal of Food Biochemistry*, 43 (4), e12809. <https://doi.org/10.1111/jfbc.12809>
- [35] Madamba, P. S., & Lopez, R. I. (2002). Optimization of the osmotic dehydration of mango (*MANGIFERA INDICA* L.) slices. *Drying Technology*, 20 (6), 1227-1242. <https://doi.org/10.1081/drt-120004049>
- [36] Beaudry, C., Raghavan, G. S., Ratti, C., & Rennie, T. J. (2004). Effect of four drying methods on the quality of osmotically dehydrated cranberries. *Drying Technology*, 22 (3), 521-539. <https://doi.org/10.1081/drt-120029999>
- [37] Perera, C. O., & Alzahrani, M. A. (2021). Ultrasound as a pre-treatment for extraction of bioactive compounds and food safety: A review. *LWT*, 142, 111114. <https://doi.org/10.1016/j.lwt.2021.111114>

Cite this article as: Bouaoudia-Madi, N., Dairi, S., Aoun, O., Kadri, N., Madani, K., & Boulekbache-Makhlouf, L. (2022). Ultrasound as pre-treatment for microwave drying of *Myrtus communis* fruits: Influence on phenolic compounds and antioxidant activity. *The North African Journal of Food and Nutrition Research*, 6 (14): 126-134. <https://doi.org/10.51745/najfnr.6.14.126-134>

© 2022 The Author(s). This is an open-access article. This article is licensed under a Creative Commons Attribution 4.0 International License, which permits use, sharing, adaptation, distribution and reproduction in any medium or format, as long as you give appropriate credit to the original author(s) and the source, provide a link to the Creative Commons license, and indicate if changes were made. The images or other third-party material in this article are included in the article's Creative Commons license, unless indicated otherwise in a credit line to the material. If material is not included in the article's Creative Commons license and your intended use is not permitted by statutory regulation or exceeds the permitted use, you will need to obtain permission directly from the copyright holder. To view a copy of this license, visit <http://creativecommons.org/licenses/by/4.0/>.