Effect of Ultrasound on Henna Leaves Drying and Extraction of Lawsone: Experimental and Modeling Study

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Abstract: The effect of drying temperature and the application of ultrasound on drying kinetics of *Lawsonia inermis* (henna) leaves and the extraction of lawsone from the dried samples was addressed. Indeed, henna leaves were dried with and without the application of ultrasound (21.7 kHz, 30.8 kW/m^3) at 40, 50 and 60 °C with a constant air velocity (1 m/s). As expected, both the increase of temperature and the application of ultrasound decreased the drying time and increased the rate of extraction of the lawsone. The values of the effective diffusion coefficients obtained were used to quantify this influence showing the value increases with higher drying temperature and the application of ultrasound. Moreover, the influence of temperature was quantified by the estimation of the activation energy from an Arrhenius-type equation (46.25 kJ/mol in the case of drying without ultrasound application and 44.06 kJ/mol in the case of ultrasonically-assisted drying). Regarding the influence of studied variables on lawsone extraction yield, the higher is the temperature, the lower is the yield, probably linked with lawsone degradation reaction due to thermal treatment. On the contrary, the application of ultrasound improved the extraction yield mainly at the lower drying temperature tested of 40 °C.

Keywords: activation energy; effective diffusivity; lawsone; *Lawsonia inermis*; ultrasound

1. Introduction

The medicinal and aromatic plants of Algeria, in particular from the southwest of the country, have attracted the attention of several researchers and scientists from different areas, such as botany, biochemistry, agri-food, chemistry and pharmaceutical field. Most of the published studies [1,2] have been focused on the chromatographic determination of the essential oil content of these plants because of their therapeutic uses. In this sense, *Lawsonia inermis* L. is an important plant widely consumed in Algerian society and worldwide. This plant grows mostly in the southern region of Algeria and is known as henna. It is characterized by a height of up to 2–3 m at the end of plant life, consisting mainly of dark green oval leaves with a length of 2–4 cm (Figure 1) that remain greenish in color for eight months [3]. In general, henna is used as a cosmetic product and is considered as an exceptional source of natural components resulting from several drug developments. Thus, henna leaves are presented as a preventive agent for treating certain diseases of the

skin, eyes and intestines [4]. More concretely, some studies showed that extracts from henna leaves have antimicrobial and antifungal actions [5] and have several industrial applications [6]. The main component identified in henna leaves extracts is the lawsone (2-hydroxy-1,4-naphthoquinone) [7,8] (Figure 2). Lawsone is used to dye the hair, fingernails, leather, silk and wool [6]. To extract this active substance, the fresh leaves of henna have to be previously dried. Traditionally, the leaves of henna are dried using solar energy. This methodology involves very long drying time and depends on the weather conditions, resulting in an important loss in the quality of the final product. Nowadays, drying is carried out at industrial scale mainly using convective hot air drying.

Figure 1. (**a**) *Lawsonia inermis* fresh leaves; and (**b**) leaves powder.
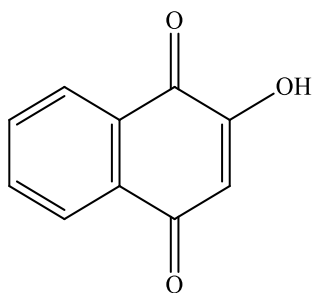

Figure 2. Structure of the Lawsone molecule.

This allows the standardization of the production and avoids problems, such as contamination by dust particles or insects [9]. As the drying process takes a long time, the conventional way to reduce this factor is to use high drying temperatures. However, this can influence the quality of the dried product and affect other characteristics such as color, texture, ascorbic acid content (AA), total carotenoids content, antioxidant properties, shrinkage and rehydration capacity [9–11].

The low efficiency of convective drying and its relatively high operating cost have made this process a subject of research to improve its results. Consequently, different of alternative technologies, such as microwaves or infrared, have been tested [11,12] with the aim of improving both the kinetics of the drying process and the quality of the final product. In this sense, high-intensity ultrasound has been found to be an efficient way to intensify convective drying of different food products and plants [13,14]. These high intensity ultrasonic waves can cause a rapid series of alternating compressions and expansions in the same way as a sponge when it is compressed and released several times (sponge effect). The sponge effect caused by the application of ultrasound may be responsible for the creation of microscopic channels in porous materials. In addition, ultrasound produces cavitation in liquid media, which can be useful in removing strongly bound moisture. At the interphases, ultrasound can generate so-called sonic wind, which reduces the diffusion boundary layer and increases the convective mass transfer [9,15]. All these effects could allow decreasing the drying time and contribute to an improvement in the quality of the product [9,16,17]. Thus, as a general rule, it can be stated that the application of ultrasound

at moderate temperatures does not produce negative effects on the quality of the product and contributes to an improvement in some quality characteristics [9,16,17]. However, this fact strongly depends on the product under consideration [9,18].

Thus, the aim of this study was to address the influence of the application of ultrasound on the drying kinetics of henna leaves and the properties of the main active component, lawsone, extracted from the henna leaves.

2. Materials and Methods

2.1. Drying Experiment

Henna leaves were cultivated in the wilaya of Bechar (southwestern region of Algeria). After the harvest, the leaves were transported from Algeria to Spain under ambient conditions; the temperature did not exceed 25 °C and there was low humidity during the travelling day. In the Agri-Food Process Analysis and Simulation Laboratory (ASPA) of the Food Technology Department, Universitat Politècnica de València, Spain, the samples were stored in plastic bags (Ziploc) at 5 ± 1 °C until the drying experiments were performed.

Drying was carried out in an ultrasonically-assisted convective dryer previously described [10,11]. This equipment is a laboratory-scale dryer modified to apply power ultrasound, with automatic control of the air temperature and velocity and provided with an automatic sample weighing system. This device includes a cylindrical vibrating radiator driven by a piezoelectric transducer (21.7 kHz), which generates a high-intensity ultrasonic field in the air medium, where the samples are placed. A high-power ultrasound generator, an impedance matching unit and a digital power meter (WT210, Yokogawa Electric Corporation, Japan) regulate and measure the electrical parameters of the acoustic signal (voltage, intensity, phase, frequency and power). The air parameters (velocity and temperature) were controlled through a PID algorithm, and a PC supervised the entire drying process. [19] The drying experiments were carried out at different air temperatures (40, 50 and 60 °C) without (Air) and with ultrasound application (Air + us; 21.7 kHz, 20.5 kW/m^3). In every run, a constant air velocity of 1 m/s was applied, and the process was stopped when the samples lost 75% of their initial weight. Each drying condition was tested at least in triplicate.

2.2. Drying Kinetics

The evolution of the moisture content of the samples during drying was determined by weighing the henna leaves at different drying times and the determination of the initial moisture content of fresh samples [20]. The equilibrium moisture content of the henna leaves was experimentally obtained by placing the henna samples in the drying conditions for a very long time. This time was enough to have no difference in sample weight for 2 h. The obtained value was compared with that previously reported by Bennaceur et al. [8]. The moisture content of the samples was then expressed in a non-dimensional way using Equation (1) [20]

$$\mathbf{MR} = \frac{\mathbf{X(t)} - \mathbf{X_{eq}}}{\mathbf{X_0} - \mathbf{X_{eq}}} \tag{1}$$

The drying rate (**Dr**) of henna leaves was calculated using the following equation [21]:

$$\mathbf{Dr} = \frac{\mathbf{X_{t+dt}} - \mathbf{X_t}}{\mathbf{dt}} \tag{2}$$

where **MR** is the dimensionless moisture content, **X(t)** is the moisture content after a drying time (**t**) (kg water/kg dry matter, d.m.), **X$_0$** is the initial moisture content of samples (kg water/kg d.m.) and **X$_{eq}$** (kg water/kg d.m.) is the equilibrium moisture content.

2.3. Lawsone Extraction and Separation

The leaves of henna, which were dried at different temperatures (40, 50 and 60 °C) with and without ultrasound application, were vacuum packaged and stored in the darkness

until processing. Each sample was extracted with methanol using a Soxhlet apparatus (laboratory of Chemistry, University of Béchar. The extraction was completed after 10 h [22]. High-performance liquid chromatography (HPLC) experiments were carried out. We employed an i-series LC-2030 (Shimadzu, Tokyo, Japan), equipped with an automatic injector of 1–100 μL sample loop and double pump system with a vacuum degassing unit. Chromatographic data were acquired, stored and analyzed by the LC Lab solution software. All parameters were deeply explored to optimise the HPLC separation.

The extraction yield ($\mathbf{R_e}$) was calculated according to Equation (3):

$$\mathbf{R_e} = \frac{\mathbf{M_L}}{\mathbf{M}} \times 100 \tag{3}$$

where $\mathbf{R_e}$ is the extraction yield and $\mathbf{M_L}$ is the weight

3. Mathematical modeling

3.1. Determination of Effective Moisture Diffusivity

The second diffusion Fick law (Equation (4)) has been widely and successfully used to describe the drying process of most biological products [23] which present a unidimensional moisture transport. In this case, it is considered that the transfer of moisture during drying is controlled only by internal diffusion.

$$\frac{\partial \mathbf{X}}{\partial \mathbf{t}} = \mathbf{D_{eff}} \, \frac{\partial^2 \mathbf{X}}{\partial \mathbf{x}^2} \tag{4}$$

where $\mathbf{D_{eff}}$ is the effective diffusion coefficient (m^2/s), t is the drying time (s) and $\mathbf{x}$ is the moisture transport direction. To integrate this equation, the following boundary and initial conditions were assumed:

- Uniform initial moisture content: $\mathbf{X(x, 0)} = \mathbf{X_0}$
- Symmetry of moisture transport: $\frac{\partial \mathbf{X}}{\partial \mathbf{x}}/_{\mathbf{x}=0} = 0$
- External resistance to mass transport negligible, which means that the equilibrium moisture content at the surface is only achieved at the beginning of the drying process: $X(L, t) = X_{eq}$

L represents the half thickness of the henna leaves. With these assumptions, the integrated solution for the whole volume of sample of Equation (4) is the one proposed by Crank [24] (Equation (5)):

$$\mathbf{MR} = \frac{\mathbf{X(t)} - \mathbf{X_{eq}}}{\mathbf{X_0} - \mathbf{X_{eq}}} = \left[\sum_{\mathbf{n}=0}^{\infty} \frac{8}{\pi^2 (2\mathbf{n}+1)^2} \mathbf{e}^{\left(-\frac{(2n+1)\pi^2 D_{eff}\, t}{4L^2}\right)} \right] \tag{5}$$

$$\mathbf{MR} = \frac{\mathbf{X(t)} - \mathbf{X_{eq}}}{\mathbf{X_0} - \mathbf{X_{eq}}} = \frac{8}{\pi^2} \mathbf{e}^{-\frac{\pi^2 D_{eff}\, t}{4L^2}} \tag{6}$$

Equation (6) can be expressed in logarithmic form as:

$$\mathbf{lnMR} = \mathbf{ln}\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 \mathbf{D_{eff}}\, \mathbf{t}}{4\mathbf{L}^2}\right) \tag{7}$$

Then, $\mathbf{D_{eff}}$ can be easily calculated from the slope of the relationship between MR and drying time (Equation (8)):

$$\mathbf{slope} = \left(\frac{\pi^2 \mathbf{D_{eff}}}{4\mathbf{L}^2}\right) \tag{8}$$

3.2. Activation Energy

The influence of the drying temperature on the D_{eff} can be quantified by the activation energy (E_a) which can be calculated by using an Arrhenius-type equation [25] as follows:

$$D_{eff} = D_0 \cdot e^{\left(-\frac{E_a}{RT}\right)} \tag{9}$$

where D_0 is a pre-exponential factor of the Arrhenius equation (m^2/s), E_a is the activation energy (kJ/mol), T is the drying temperature (K) and R is the constant of ideal gases (kJ/molK).

4. Results and Discussion

4.1. Experimental Drying Kinetics

The initial moisture content of henna leaves was 2.33 kg water/kg d.m. The drying air temperature affected the experimental drying kinetics (Figure 3a). As expected, it was observed that the increase of the air temperature allowed decreasing the drying time. Thus, the time needed to achieve a moisture content of 1 kg water/kg d.m. was 3.47 ± 0.63 h at 40 °C, 1.97 ± 0.05 h at 50 °C and 1.19 ± 0.05 h at 60 °C. Thus, increasing the drying temperature from 40 to 60 °C reduced the drying time by 35%.

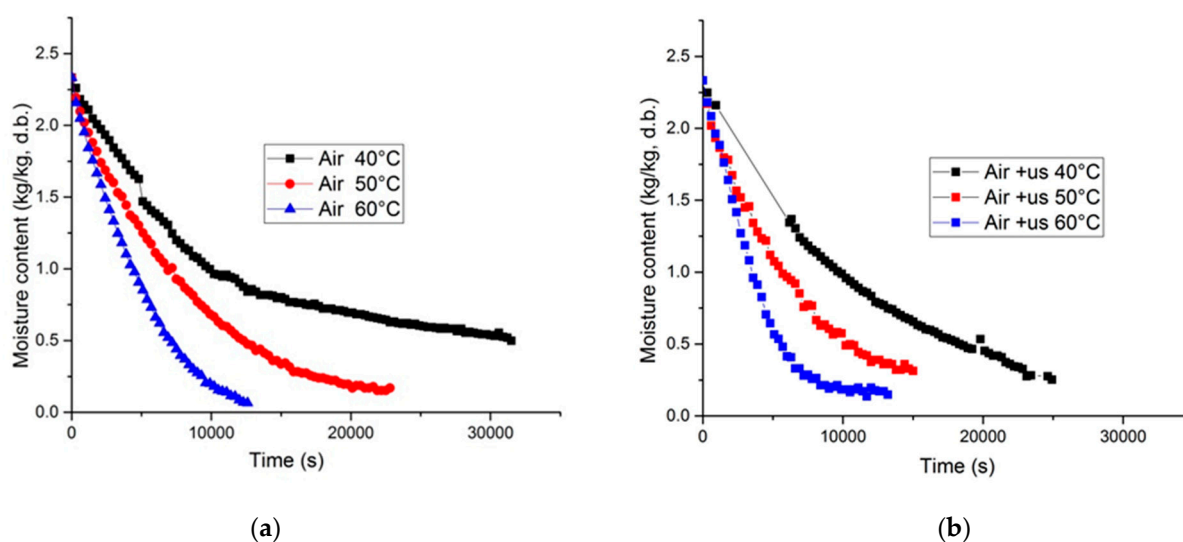(**a**)   (**b**)

Figure 3. Experimental drying kinetics of the henna leaves determined at different temperatures (40, 50 and 60 °C): (**a**) without ultrasound application (Air); and (**b**) with ultrasound application (Air + us; 20.5 kW/m³; 21.7 kHz).

The application of ultrasound during convective drying significantly increased the drying kinetics of the henna leaves (Figure 3b). For example, at 40 °C, the drying time in experiments carried out with ultrasound was 40% lower than those carried out without ultrasound (Table 1). Ultrasound can improve moisture removal by affecting both the internal movement of water inside henna leaves producing the "sponge effect" and the transport to moisture from the henna leaves surface to the drying air by the micro-stirring at interfaces generated by the so-called ultrasonic wind [13].

Table 1. Drying of henna leaves (1 m/s) at different temperatures, without and with ultrasound (21.7 kHz, 20.5 kW/m^3). Drying time needed to reach a final moisture content of 01 kg of water/kg of dry matter (mean $\pm$ SD).

Temperature (°C)	Ultrasound Application	Time (h)
40	No	3.47 $\pm$ 0.63
50	No	1.97 $\pm$ 0.05
60	No	1.19 $\pm$ 0.05
40	Yes	2.64 $\pm$ 0.13
50	Yes	1.64 $\pm$ 0.17
60	Yes	1.11 $\pm$ 0.19

In drying operations, it is interesting the study of the variation of drying rate versus the moisture content evolution, which called the Krischer's curve (Figure 4)
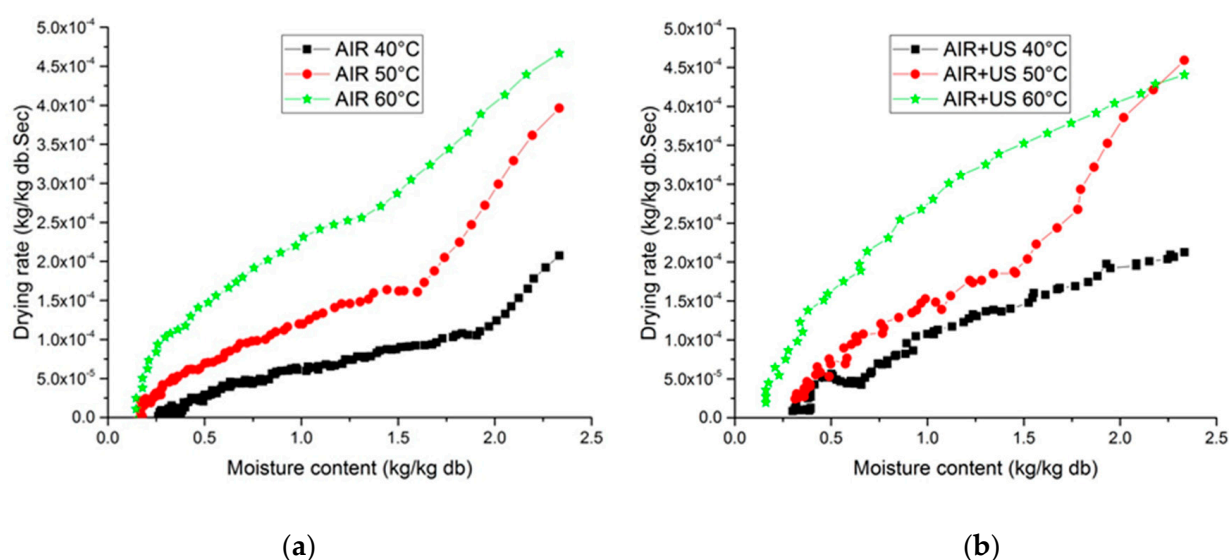

(**a**)   (**b**)

Figure 4. Drying rate curves of leaves henna vs. moisture content at different temperatures (40, 50 and 60 °C): (**a**) without ultrasound application (Air); and (**b**) with ultrasound application (Air + us; 20.5 kW/m^3; 21.7 kHz).

In this can, only the falling rate period was observed in all the experiments. The drying rate increased with increasing temperature. During the drying process of fresh henna leaves, the ratios between drying speeds without the application of ultrasound (AIR) for temperatures 50 and 60 °C and temperature 40 °C at moisture content of 1 kg water/kg d.m were 20,052% and 386%, respectively. This indicates the thermal effect observed on the drying rate. In the case of experiments carried out without ultrasound application, two stages were observed: the slope of the relation between drying rate and moisture content above a specific moisture content was significantly larger than the one below this moisture content (Figure 4a). This is because, at the beginning of drying, the free moisture content is eliminated. In this stage, the decrease of drying rate with the decrease of moisture content is fast. This specific moisture content depended on the drying temperature applied, being lower at higher drying temperature (1.9 kg water/kg d.m. at 40 °C, 1.6 kg water/kg d.m. at 50 °C and 1.2 kg water/kg d.m. at 60 °C). The higher is the temperature, the greater is the energy in the system, which makes the water movement easy. Below the specific moisture content, the remaining moisture is bound to the henna matrix. In this situation, the decrease of the drying rate with the reduction of moisture slowed down.

The application of ultrasound affected the evolution of drying rate with the moisture content. The drying experiments carried out with ultrasound showed an increase in the

drying rate compared with those performed without ultrasound. Thus, the ratios between drying speeds of the two drying methods (Air + us/Air) were 180.60%, 127.50% and 121.64% for 40, 50 and 60 °C, respectively, at moisture content of 1 kg water/kg d.m.

Regarding the evolution of drying rate, in the case of ultrasonic-assisted drying experiments, the evolution was continuous in the whole range of moisture content, and only at the drying temperature of 50 °C it was possible to identify the two stages identified in the drying carried out without ultrasound (Figure 4b). This could be due to the ultrasound effects can making the movement of water inside the samples easy and thus no difference in bounded and unbounded water removal appeared.

4.2. Effective Diffusivity and Activation Energy

To quantity the influence of temperature and ultrasound application on the drying kinetics, experimental moisture content evolution was modeled using Equation (5). The values of the identified effective diffusion coefficient (D_{eff}) and the correlation coefficient at different temperatures (40, 50 and 60 °C), with and without the application of ultrasound, are shown in Table 2. The correlation coefficient was above 0.98 in every experiment, which indicates that the model adequately fit the experimental data. The D_{eff} values obtained ranged between 0.7094×10^{-9} and 2.5547×10^{-9} m^2/s in the case of conventional drying experiments and 1.0234×10^{-9} and 2.8164×10^{-9} m^2/s in the case of ultrasonically-assisted ones. These values are in the same range as others given in the literature [26,27].

Table 2. Effective diffusivity (D_{eff}) identified for the drying of henna leaves at different temperatures, without and with ultrasound (21.7 kHz, 20.5 kW/m^3) (mean ± SD).

Temp (°C)	Air		Air + us	
	D_{eff} (m^2/s) 10^{-9}	r	D_{eff} (m^2/s) 10^{-9}	r
40	0.7094 ± 0.2253	0.9835	1.0234 ± 0.0398	0.9929
50	1.5933 ± 0.1218	0.9920	2.0882 ± 0.9859	0.9887
60	2.5547 ± 0.4922	0.9930	2.8164 ± 0.3576	0.9834

The application of ultrasound increased the D_{eff} identified at the temperature tested. The ultrasound effects could contribute to accelerate the water movement and removal from the henna leaves. Regarding the temperature, it was observed than the the D_{eff} increased with the drying temperature. To evaluate the influence of drying temperature in drying kinetics, an Arrhenius-type equation was used to calculate the activation energy. Thus, the natural logarithm of the identified values of D_{eff} was represented as a function of the temperature inverse, and, from the slope of this relation, the activation energy was calculated.

The values obtained were 62.23 ± 1.78 and 43.83 ± 1.42 kJ/mol for drying without and with the application of ultrasound, respectively (correlation coefficients of 0.9840 and 0.9906, respectively). These values are similar to those reported in the literature [26,27]. Therefore, the influence of drying temperature in drying kinetics was slightly lower in the experiments carried with ultrasound application.

4.3. Influence of Drying in Lawsone Content

To confirm the presence of lawsone in our samples, we used the results obtained from the analysis by HPLC [28]. The chromatograms were developed at 293 nm and then compared with that of pure lawsone. As shown in Figure 5a,b, the peak obtained at 4.404 min (retention time) for the pure lawsone corresponds with the peak obtained at 4.413 min for the extract obtained from henna leaves dried at 40 °C without ultrasound application.
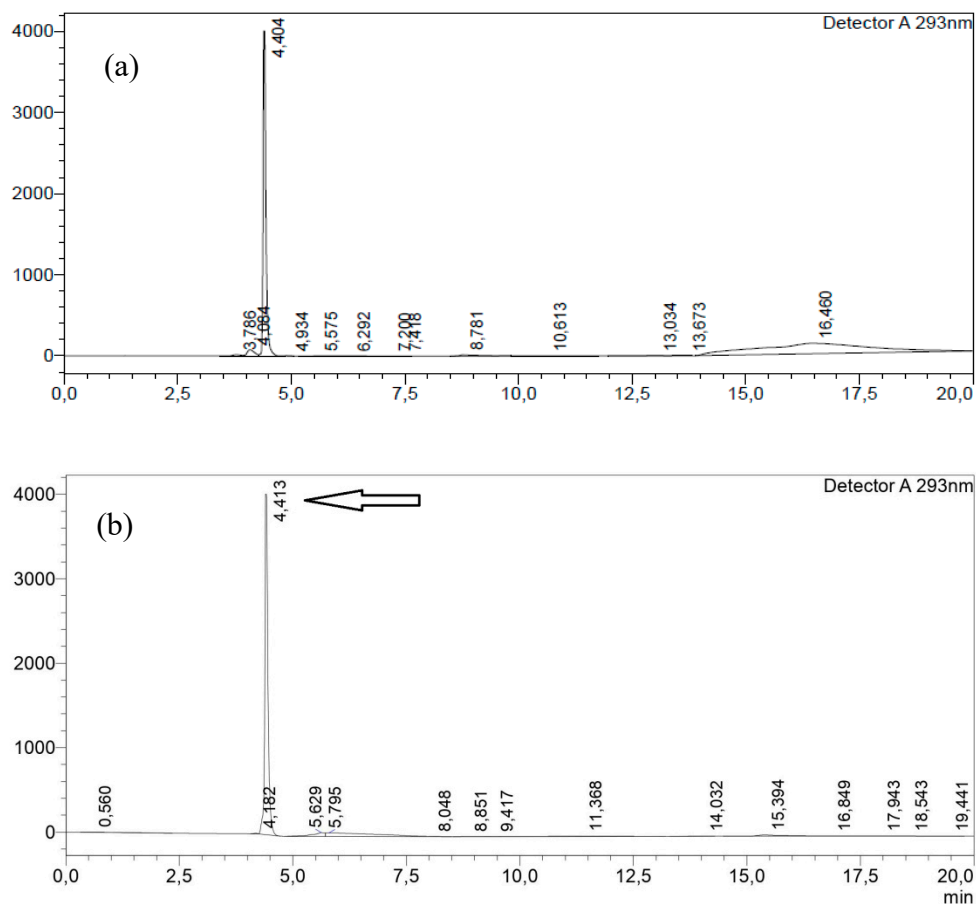
Figure 5. HPLC analysis for: (**a**) pure Lawsone; and (**b**) samples of dried leaves without ultrasound (Air) at 40 °C.

As shown in Figure 6, the drying temperature highly affected the extraction yield (Re), Thus, Re decreased with the increase of drying temperature. The higher is the air temperature, the greater is the amount of energy supplied, and this likely produces a greater degradation of the product. Therefore, the drying at low temperature of henna leaves permits obtaining a greater yield of lawsone extracts.
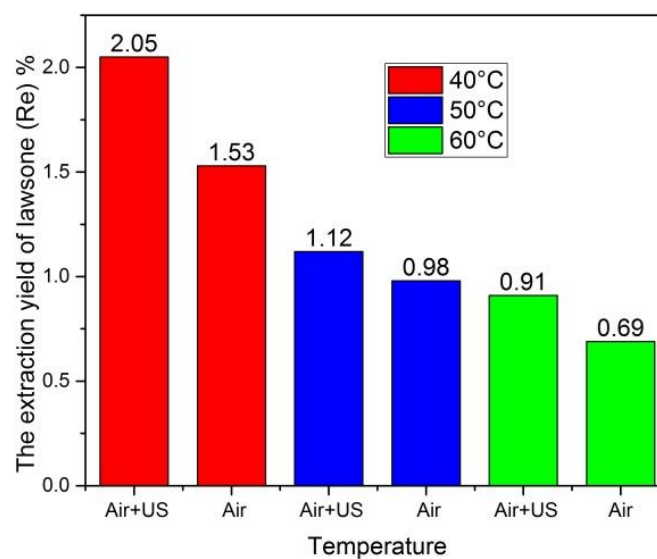

Figure 6. The extraction yield of lawsone extracted from henna leaves at 40, 50 and 60 °C without (Air) and with (Air + us; 20.5 kW/m^3; 21.7 kHz) ultrasound application.

Regarding the effect of the ultrasound application during drying, it was observed that, for all drying temperatures (40, 50 and 60 °C), the lawsone extraction yields were significantly higher in the ultrasonically-assisted dried henna leaves (Air + us) than in the conventionally dried ones (Air). This could be related with the shortening of the drying process when ultrasound is applied. Thus, the extraction yield differences were obtained in samples dried at 40 and 60 °C. In the first case, the slow drying kinetics could increase the degradation of lawsone by oxidation reactions. In this case, application of ultrasound reduced drying time and the time of exposure to the drying air, limiting oxidation reactions. In the second case, the higher drying temperature applied (60 °C) could affect the lawsone integrity. The shortening of drying process produced by ultrasound application reduced the exposure time to high temperature and, therefore, the thermal damage.

Moreover, it is known that the application of ultrasound during drying can produce some effects in the microstructure of products. Thus, the sequential compressions and expansions induced by ultrasonic waves can affect the integrity of cell walls or produce the generation of micro-channels in the material [13]. These effects can improve the extraction of lawsone from henna leaves and increase the yield.

5. Conclusions

The influence of drying temperature and ultrasound application in the drying kinetics of henna leaves was addressed. In the range of temperature tested, the higher is the temperature, the faster is the drying. The impact of the temperature and application of the ultrasound on the diffusivity coefficient temperature followed the same trend. However, the relationship between temperature and extraction yield of lawsone, the most interesting compound of henna leaves, was the opposite: the greater is the temperature, the lower is the extraction yield, likely because of thermal degradation. The application of ultrasound increased the drying kinetics as well as the extraction yield of lawsone. Therefore, ultrasonically-assisted drying of henna leaves can be considered to both reduce drying temperature to save energy and increase the yield of the extraction of lawsone. Ultrasonic-assisted drying provided a good effect on the drying process, but the extent of ultrasonic improvement depended largely on process variables, such as air velocity and range of ultrasound frequencies. The application of ultrasound technology will in some way affect the quality of food, including physical and chemical attributes. In general, the application of ultrasound can decrease water activity and improve product quality.

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Conflicts of Interest: The authors declare no conflict of interest.

Abbreviations

D_0	Pre-exponential factor of the Arrhenius equation [m^2/s]
D_{eff}	Effective diffusivity [m^2/s]
E_a	Activation energy [kJ/mol]
L	Half-thickness of el Henna's leaves [m]
M	Mass of the dry sample [g]
MR	Dimensionless moisture content [-]
M_L	Lawsone mass [g]
Re	Yields of lawsone [-]
r	Correlation coefficient [-]
t	Time
T	Temperature [°C or K]
x	Mass transport direction [m]
X	Moisture content
X_{eq}	Equilibrium moisture content [kg/kg % d.b]

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