

Article

Optimized Solid–Liquid Separation of Phenolics from Lavender Waste and Properties of the Dried Extracts

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Abstract: Lavender distillation produces huge quantities of solid waste yearly. This waste is usually discarded, resulting in serious environmental issues. However, it still contains residual essential oil and other bioactive compounds. This research reports on the development and comparison of optimized solid–liquid separation methods, i.e., microwave- (MAE) and ultrasound-assisted extraction (UAE) of phenolic compounds from lavender distillation waste. The optimal pretreatment conditions, such as waste moisture content and particle size, were also determined. The extracts were spray- or freeze-dried and the resulting powders were characterized for their physicochemical properties. The majority of the original phenolic compounds in lavender were found in the leachate fraction after distillation (61%), whereas 43% was found in the solid waste. Drying of the solid waste before extraction affected the process efficiency. UAE led to a higher phenolic content and greater antioxidant properties compared to MAE. Drying (spray or freeze) the extracts did not significantly affect their phenolic content, whereas the use of maltodextrin as a drying agent improved the drying process yield, especially when using the freeze-drying method. It is concluded that valorization of lavender distillation wastes can be achieved via an integrated process consisting of a green extraction method and a consequent drying process that results in a stable bioactive powder.

Keywords: extraction; lavender; phenolics; waste; spray drying; freeze-drying; microwave; ultrasound

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1. Introduction

Lavender is renowned for its aromatic and versatile nature, being widely acknowledged for both its therapeutic and fragrant qualities. It originates from the Mediterranean areas of Europe and Northern Africa and is cultivated in several European, South American, North African, and southwest Asian countries and Australia. There are more than 32 species of lavender and abundant subspecies and varieties. However, only three of them are widely used in the production of essential oil, common lavender (*Lavandula angustifolia* Mill.), lavender aspic (*Lavandula spica* L.), and lavandin (*Lavandula hybrida*) [1,2].

With a rich history of use in traditional medicine, cosmetics, and aromatherapy, lavender essential oil is a valuable natural product renowned for its soothing and calming effects. The high demand for lavender essential oil in various industries, including perfumery, personal care products, and complementary medicine, has led to a growing interest in efficient methods for its extraction. Distillation is one of the most widely employed methods for lavender essential oil extraction, and its effectiveness is contingent

upon the choice of distillation equipment, operational parameters, and the quality of the lavender material.

The waste generated during lavender distillation, commonly referred to as lavender distillation waste, consists of several components, each with its own potential for valorization or responsible disposal. The liquid by-products of the distillation are wastewaters named hydrolats (or hydrosols) and leachates. Hydrosol is the aqueous by-product resulting from the condensation of steam that carries essential oil constituents and is often used in cosmetics, skincare products, and aromatherapy as a fragrant and soothing water, thereby reducing waste and providing added value. Leachates are aqueous mixtures containing water-soluble compounds with high molecular weights, which are extracted from the plant by heating but do not undergo distillation.

In addition, there are lavender plant residues (stems, leaves, and flowers) that are left as a solid waste after distillation. According to Vasileva et al. [3], the low amount of essential oil in the lavender (0.8–1.3%) leads to huge quantities of solid waste produced yearly. These wastes are usually discarded in nearby places, resulting in serious environmental issues. However, they still contain residual essential oil and other bioactive compounds and can potentially be valorized in the following ways:

- Use as a source of biomass for bioenergy generation, such as the production of bio-fuels or biochar [1,4–7].
- Conversion into mulch, compost, or soil conditioner, contributing to soil health in agriculture [8–10].
- Extraction of bioactive compounds for secondary product manufacture [1,11,12].

Lavender solid waste has been determined as a rich source of phenolic acids (p-coumaric, neochlorogenic, caffeic, and rosmarinic acid) and flavonoids (quercetin-3- β -glucoside, quercetin and mircetine) with antioxidant and antimicrobial activities [3,12]. Méndez-Tovar et al. [11] studied the total phenolic content and antioxidant activity of *Lavandula latifolia* waste produced after removing the essential oil by distillation, whereas total phenolic and flavonoid contents of lavender leafy stalks (*Lavandula angustifolia*) were identified by Adaszyńska-Skwirzyńska et al. [13]. Recently, Truzzi et al. [14] characterized the by-products from the steam distillation of *Lavandula angustifolia* Mill. (LA) and *Lavandula x intermedia* Emeric ex Loisel (LI) aerial parts for their total polyphenol and flavonoid contents. Despite this, very few studies are available on the process of extraction of phenolic compounds from lavender solid waste. Ivanov et al. [15] studied the phenolic content in extracts (with methanol, ethanol, and acetone) obtained by various techniques (maceration and ultrasound-assisted extraction) from lavender solid waste. Recently, Turrini et al. [16] used pulsed ultrasound-assisted extraction as a sustainable technology to extract lavender solid waste using a mixture of ethanol and water.

Microwave-assisted extraction (MAE) stands out as a widely adopted alternative method for extraction. Its advantages encompass shortened extraction duration and reduced solvent consumption. The accelerated process and improved extraction rates in MAE are reported to stem from the synergistic effect of thermal and mass transport phenomena [17]. Microwave treatment has been extensively applied for the isolation of phenolics from diverse plant materials, making MAE a promising technique. To our best knowledge, there are no studies on the MAE of phenolics from lavender waste. In addition, according to Zheng et al. [18], optimizing the extraction parameters is crucial for comprehending the interaction among independent factors and determining the optimal combination of extraction parameters. Additionally, the efficiency of MAE relies on several variables that may not be universally applicable to all plant materials due to the diverse array of bioactive phytochemicals present. Therefore, selecting and optimizing processing conditions should be tailored to the specific matrix used, considering the desired response.

Ultrasound-assisted extraction (UAE) also stands out as a sustainable alternative that requires a moderate investment of solvent and energy [19]. UAE is based on the principle

of acoustic cavitation, which is capable of damaging the cell walls of the plant matrix and favors the release of bioactive compounds [20]. Several research works have been recently published on the use of UAE applied to the extraction of phenolic compounds from a number of plants such as myrtle (*Myrtus communis* L.) [21], pomegranate peel (*Punica granatum* L.) [22], maqui (*Aristotelia chilensis* (Mol.) Stuntz) [23], black chokeberry (*Aronia melanocarpa* L.) [24], and lavender (*Lavandula angustifolia* Mill.) [16], among others. The efficiency of this method for phenolic compound extraction may also be altered by many factors such as time, temperature, solvent, pH, solvent–sample ratio, and ultrasound amplitude [25]. As a result, the optimization of these parameters is important to obtain the maximum yield of the target compounds. To our best knowledge, as far as lavender waste is concerned, only Ivanov et al. [15] and Turrini et al. [16] have employed UAE to extract phenolic compounds.

In addition, polyphenol extracts can easily deteriorate when exposed to oxidants, light, and heat, due to the existence of unsaturated bonds in their structure. As a result, solid forms of extracts are being increasingly used since they have numerous advantages over fluid extracts, such as better stability, cheaper transport and storage, and the possibility of increased concentrations [26]. However, no work has been conducted on the drying of lavender waste extract.

The waste generated during lavender distillation presents an opportunity for sustainable valorization and resource recovery. Responsible waste management practices and the development of innovative processes for utilizing this waste can significantly reduce the environmental impact of lavender distillation and contribute to the sustainability of the lavender industry. Additionally, it aligns with the principles of the circular economy, where waste materials are transformed into valuable resources rather than being disposed of as a burden to the environment.

Thus, the objective of the present work is to develop and optimize an effective alternative and “green” method for the extraction of phenolics from lavender solid waste focusing on MAE and UAE. The pretreatment conditions (solid waste moisture content and particle size), as well as the extraction parameters for both MAE and UAE were investigated and optimized for the maximum phenolics yield. The obtained extract is characterized for its antioxidant capacity and is stabilized by spray- and freeze-drying. The main properties of the produced powders (moisture content, water activity, density, hygroscopicity, and total phenolic content) are determined. The obtained dry extract can be used as a functional ingredient in fortified foods to supplement them with biofunctional metabolites.

2. Materials and Methods

2.1. Raw Material

The aerial parts of *Lavandula stoechas* were kindly provided by local producers in Rodolivos (Rodolivos IKE, Serres, Greece). They were harvested in the full-bloom stage in June 2022. The plant material had a moisture content of about $18 \pm 1\%$ wet basis and was cut into 2–3 cm pieces. Then, it was packed and placed in a cool place.

2.2. Hydrodistillation

Thirty (30) g of lavender was submitted to hydrodistillation using a Clevenger-type apparatus (LG-11125-102, Wilmad-LabGlass, Warminster, PA, USA) and extracted with 600 mL of water. The distillation was carried out using an electric heating mantle (Model 655, Nahita Ltd., London, UK) for 105 min, resulting approximately in a 1.5 mL essential oil yield. Further distillation was observed to have a minimal effect on the oil yield. The distillation products and by-products were as follows:

- Essential oil (EO)
- Solid waste (SW): the residual plant material of lavender flowers
- Liquid waste or leachate (LW): wastewater in the glass flask

- Hydrolate or floral water (Hydro): condensed water vapors

2.3. Distribution of Phenolics in the Hydrodistillation Products and By-Products

For the analysis of the total phenolic content in the hydrodistillation products, lavender flowers, solid waste (SW), and essential oil (EO) needed to undergo an extraction procedure prior to measuring their total phenolic contents, whereas the liquid waste (LW) and hydrolate (Hydro) fractions did not need any pretreatment. Both the flowers and solid waste fractions first underwent grinding (size 1–5 mm). Extraction was conducted with 130 W of maximum nominal output power and 20 kHz of frequency using a VCX-130 Sonics and Materials (Danbury, CT, USA) sonicator equipped with a Ti–Al–V sonoprobe (13 mm). Two T-type thermocouples were submerged in the extraction solution, while the sample container was kept in a thermostat-controlled water bath. The temperature was maintained by periodically adding ice. The selected operating conditions were based on a previous work (solvent: aqueous ethanol solution 50%, *v/v*, extraction temperature: 35 °C, solvent-to-solid ratio: 33 mL/g, pulse on/off: 7/6, sonication time: 15 min, amplitude level: 60%) [17].

As far as the pretreatment of essential oil (EO) is concerned, 5 g of essential oil was mixed with 10 mL of aqueous ethanol solution 50% *v/v* and 0.2 g Tween 80, followed by stirring for 5 min. Extraction was conducted in an ultrasound water bath at 35 kHz frequency, 860 W maximum nominal output power (Bandelin electronic, Model DL 512 H, Berlin, Germany), 40% amplitude, and 35 °C temperature for 30 min. The sample was then stirred for 5 min and centrifuged at 3000 rpm for 20 min. After centrifugation, the ethanolic fraction was isolated and the whole process was repeated once more in the oil fraction. Finally, after two repetitions, the ethanolic fractions were the fractions measured for total phenolics.

2.4. Pretreatment Parameters of Hydrodistillation Solid Waste

To study the effect of solid waste moisture content on extraction yield, solid waste samples were dried at 40 °C to various moisture contents (5, 8, and 15%). The solid waste was filtered to remove the residual water amount and dried in a tray dryer (Gallenkamp PLC, Model BR185HNI/NCC, Leicester, England) with an air velocity of 1.2 m/s, at a temperature of 40 °C. The samples were prepared in 23.0 cm × 17.2 cm dishes. Three replicate runs were conducted. The drying data were obtained by periodic weighing of the samples with a balance (PCB 1000-2, KERN & Sohn GmbH, Balingen, Germany). The initial moisture content was determined by the vacuum oven method at 100 °C for 24 h.

In addition, the lavender solid waste was processed using three different grinding degrees to study the effect of the solid waste particle size on extraction yield. Three samples were used, an intact dried lavender waste sample (10 mm) and two samples grounded through sieves of 1 and 4 mm.

2.5. Ultrasound-Assisted Extraction (UAE) of Solid Waste

Ultrasound-assisted extraction (UAE) was conducted with an ultrasonic homogenizer (Sonopuls HD 4100, Bandelin, Berlin, Germany). One (1) g of dried lavender solid waste was mixed with 18 mL of ethanol–water solvent (1:1) and the solution was extracted with an ultrasound amplitude level of 40% for 10 min. The temperature was maintained by periodically adding ice. The above operating conditions were determined in optimization experiments presented in the Supplementary Material (Figures S1 and S2, Table S1). The extracts were then filtered and stored at −18 °C until further analysis.

2.6. Microwave-Assisted Extraction (MAE) of Solid Waste

Microwave-assisted extraction was conducted with a Multiwave closed microwave system (B30MC030A, AntonPaar, Graz, Austria), equipped with a standard configuration and a rotor for 6 sample vessels type H (50 mL quartz tubes; operating pressure 75 bars).

A sample of dried solid waste (SW) was mixed with 20 mL solvent to produce different solvent/SW ratios. The extracts were collected at 4 min. The objective was to determine the optimum conditions in terms of microwave power (P), solvent/SW ratio (L/S), and solvent concentration (S) for the extraction of phenolic compounds with two different solvent types (ethanol and methanol). Since this work studies the effect of three factors for 2 types of solvent, a central composite design (CCD) would result in 40 experiments. Thus, to reduce the number of experiments and not to be restricted to the predefined levels of factors for the design of experiments as in CCD, an optimal design was used. An I-optimal design was selected to determine the effects of the above parameters (P , L/S , S) on the extraction yield for the two solvents (Table 1).

Table 1. I-optimal experimental design for the MAE of lavender solid waste for each different solvent (16 experiments for ethanol and 16 experiments for methanol).

Experiment No.	Microwave Power (P , W)	Solvent Concentra- tion (S , % v/v Aqueous Ethanol or Methanol)	Liquid/Solid Ratio (L/S , mL/g)
1	100	100	30/1
2	800	0	50/1
3	800	100	34/1
4	500	70	34/1
5	300	70	20/1
6	100	0	46/1
7	300	0	34/1
8	300	70	40/1
9	300	30	30/1
10	100	100	34/1
11	300	70	30/1
12	100	10	50/1
13	300	30	40/1
14	100	100	46/1
15	300	100	40/1
16	300	100	40/1

2.7. Characterization of Extracts

2.7.1. Total Phenolic Content (TPC)

The total phenolic content was determined for the distribution of phenolics both in the distillation products (Section 2.3) and in the UAE and MAE extracts of solid waste (Section 2.5). The quantification of the total phenolics was carried out following the Folin–Ciocalteu spectrophotometric assay [17]. Extraction Yield (Y), denoted in mg GAE/g, was determined as the ratio of the total weight of gallic acid equivalents (GAE) extracted to the mass of the used sample.

2.7.2. Antioxidant Activity (AA)

The in vitro antioxidant activity of extracts was examined by employing the DPPH• scavenging activity [27] and the ferric reducing antioxidant power (FRAP) assays [28].

2.8. Drying of Extracts

Extracts prepared in the optimum conditions (MAE and UAE) were concentrated to a solids concentration of 10% (w/w) in a rotary vacuum evaporator (Model R 114, Buchi Laboratoriums-Technik, Flawil, Switzerland). The final extracts were stored at -18 ± 1 °C in glass bottles until the drying experiments. In the case of spray-drying, after preliminary

experiments, it was found that the addition of maltodextrin (10 DE) at a content of 10% *w/v* was necessary to increase the efficiency of the process. Therefore, the concentrated extract was mixed with maltodextrin for 24 h before being introduced into the spray-dryer.

For the spray-drying experiments, the liquid feed was spray-dried using an ADL 311S spray-dryer (Yamato Scientific Co., Tokyo, Japan) with a maximum compressed air pressure of 0.1 MPa, a feed flow rate of 3.9 mL/min, a drying air flow rate of 0.07 m³/min, and an inlet air temperature of 170 °C. The resulting outlet temperature was about 57 ± 2 °C. The powder was collected, vacuum packed, and stored until further analysis.

In the case of freeze-drying, the concentrated extract was subjected to a freezing process at a temperature of −18 °C for 48 h. Subsequently, the frozen material was transferred to a lyophilizer (model Alpha 1–4 LD Plus, Martin Christ, Osterode am Harz, Germany) that was equipped with a capacitance manometer for precise monitoring of the condenser pressure. The freeze-dryer was operated under vacuum (1.6 mm Hg) for an additional 24 h. This procedure continued until the moisture content was stabilized. During the process, the chamber was maintained at a temperature of 35 °C, whereas the ice condenser was set at −55 °C. The resulting powder was stored under refrigeration.

2.9. Characterization of Dried Extracts

The powders produced by spray-drying and freeze-drying, with and without maltodextrin, were characterized for the following main properties. All analyses were performed in triplicate.

Moisture: Drying at 70 °C in a vacuum oven was used to measure the moisture content until successive weighing produced a change of less than 0.3% [29]. The moisture content was given as 100 × kg water/kg wet material or the percent wet basis (w.b.).

Bulk density: One 15 mL graduated cylinder was filled with 2 g of powder. By dividing the mass of the powder by the volume inside the cylinder, the aerated bulk density was computed.

Hygroscopicity: On Petri plates with a diameter of 9 cm, 1 g of powder was equally distributed to provide a large surface area between the powder and the humid air at 25 °C and 76% relative humidity. The hygroscopicity was measured after 48 h and was expressed as g of absorbed water per 100 g of dry powder.

Water activity: Water activity was measured using a Rotronic HygroLab laboratory analyzer.

Total phenolic content and antioxidant activity: About 1 g of powder was added to 20 mL of water and the total phenolic content and antioxidant activity were determined according to the methods described in Section 2.7.

2.10. Statistical Analysis

To determine the significance of the effects and their interactions, analysis of variance (ANOVA) was performed. A *p*-value of less than 0.05 was considered statistically significant. Minitab (Release 15, Statistical Software, MINITAB Inc., Lock Haven, PA, USA) was used to analyze the obtained experimental data and determine the optimal experimental conditions.

3. Results and Discussion

3.1. Distribution of Phenolics for the Distillation of Lavender Flowers

The phenolic compound distribution for the different fractions mentioned in Section 2.3 is presented in Figure 1. The basis to compare the phenolic content of each fraction was the TPC of raw material. The processing of waste streams more often than not requires a drying process to remove excess water depending on the type of distillation used [16,30,31]. The initial distilled flower solid waste had absorbed high amounts of water and this was evident on the reduced residual water volume after the process and the increase

in solid material weight. Lavender flowers indeed had the highest amount of TPC at 18.05 mg GAE/g dry weight (d.w.).

For each distillation, 30 g of raw material yielded approximately 19 g of flower waste that had $43 \pm 2\%$ of the phenolic content of the raw material. Lavender flower solid residues, like most aromatic plant solid waste, were reported to be compromised by water-insoluble matter such as hemicellulose, cellulose, and lignin, along with compounds of a phytochemical nature [1,32]. The plant origin and, in particular, the distillation technique heavily affect the resulting distribution of phenolics [33]. In hydrodistillation, the water and raw material come into direct contact, enabling the leaching of soluble solids into the main volume of water. The water solubility of phenols is not originally very high but becomes greatly enhanced by the high temperatures that occur during distillation; this is because of their ability to form hydrogen bonds with water molecules, which ultimately enables their dissolution in the wastewater [34,35].

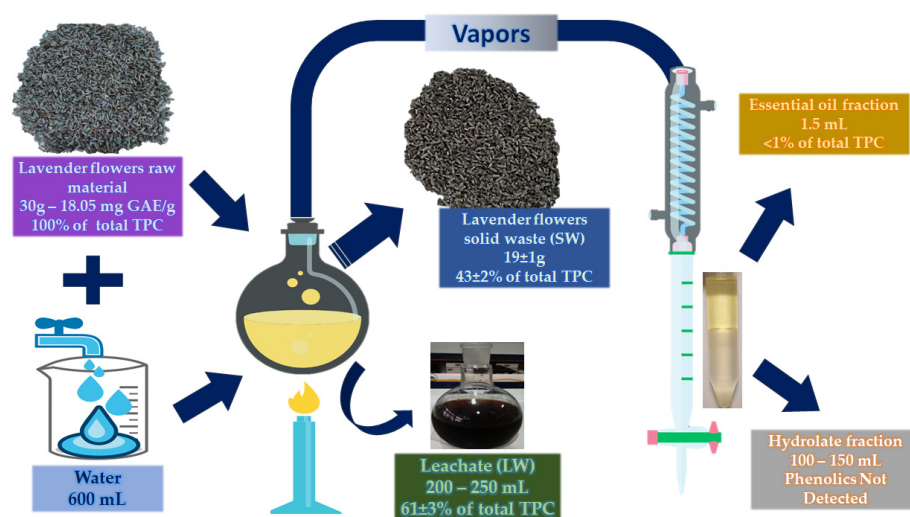


Figure 1. Distribution of total phenolic content in each fraction of the distillation procedure.

Richly infused wastewaters after the hydrodistillation process had been reported for *T. vulgaris* [36] and *H. officinalis* [37]. Wollinger et al. [38] reported that an increase in hydrodistillation time up to 2.5 h had a positive effect in continuously enriching the wastewaters of *R. officinalis* with antioxidants. Alice et al. [39] studied different *Lamiaceae* species and similarly reported a phenolics reduction in the main solid waste and an increase in wastewaters due to solubilization and degradation. Wastewaters in the current study amounted up to more than half ($61 \pm 3\%$) of the phenolics present in the raw material, which implies their affinity to water being enhanced by the distillation heating. The phenolic compounds of lavender distillation residues had already been reported to be of medium-hydrophilic nature and their optimum extraction could be augmented with the addition of solvents, which complies with the assumptions of the current study [1,15,16,31,40]. Turrini et al. [16], on the other hand, reported minimal amounts of phenolic compounds in the wastewaters compared to the flower solid waste of lavender. However, it should be noted that the distillation method used was steam distillation, which potentially minimizes the solubility of such substances that were left intact in the solid waste tissue.

The essential oil fraction showed negligible amounts of phenolic compounds. Lavender essential oil contains many compounds of a volatile nature, such as terpenes and terpenoids, and is mainly composed of the monoterpenes linalool, linalyl acetate, and *trans* β -ocymene. The phenolic compound content in the essential oils is greatly affected by the distillation method and the duration, as well as the plant tissue [30]. However, because of

the nature of the process, only phenolic compounds of a volatile and/or lipophilic nature will be mostly present in the oil. Furthermore, the oil quantity was far lower than the rest of the by-products and the major volume of phenolics was already determined in the solid and liquid wastes.

Phenolic compounds in the hydrolate fraction were not detected. In a similar manner to the essential oil, only compounds of a volatile nature are able to transverse the Clevenger apparatus and be present in the hydrolates. In the literature, the most widely known and identified compounds in the hydrolate fraction were the monoterpenes thymol and carvacrol, as well as the phenylpropene eugenol [33]. However, these substances are not present in *Lavandula angustifolia* and the hydrolates consist mostly of linalool, borneol, and linalool oxides [41]. The hydrolate residue of lavender flowers has not been examined widely. In the study by Turrini et al. [16], a similar trend was observed regarding the hydrolates in lavender steam distillation. They reported that hydrolates had really low phenolic contents, while both their antiradical activity (RSA) and flavonoid content (TFC) were not detectable.

3.2. Effect of Lavender Solid Waste Moisture and Degree of Grinding on Extraction Efficiency

The drying curve of lavender solid waste was obtained by monitoring the moisture content of the material over time under controlled drying conditions (40 °C). The drying process was analyzed to assess the variations in drying rates at different stages and to prevent over-drying, which can lead to product degradation, or under-drying, which can result in product spoilage or inefficient extraction. The initial moisture content of the sample was found to be 88.2%. From the drying curve (Figure 2), three parts can be distinguished: (i) the initial transition period, where the rate of moisture removal is slow and the curve has a slight downward slope (1–4 h), (ii) the constant drying rate phase (4–12 h), and (iii) the reduced drying rate phase, where the rate of moisture removal slows down (12–14 h).

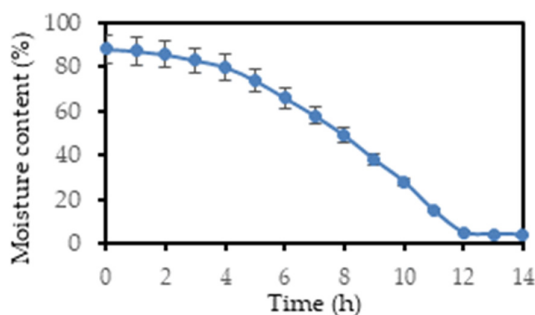


Figure 2. Drying curve of lavender solid waste during a 14 h period.

The comparison of the samples with moisture contents of 15, 8, and 5% was carried out by collecting them and conducting ultrasound extraction under the same optimized conditions. Drying enhances the extractability of active ingredients due to the destruction of the cell walls allowing cellular substances to be easily extracted. Furthermore, the lack of water also contributes to the inhibition of enzymes that can break down bioactive compounds. On the other hand, excessive drying might cause the loss of active ingredients due to prolonged exposure to the drying temperatures [42,43]. The antioxidant activity of each extract was assessed through the determination of the TPC (total phenolic content) and the use of the DPPH (2,2-diphenyl-1-picrylhydrazyl) assay, as well as the FRAP (ferric reducing antioxidant power) assay. The results (Figure 3) indicate that the drying degree of lavender affects, to some extent, the TPC and the % antioxidant capacity values, since they are significantly higher (p -value < 0.05) in the sample with moisture content of 5% and generally decrease with higher moisture contents. The FRAP assay shows slightly

better values in the 5% moisture content sample, although it is not considered statistically important (p -value > 0.05).

The lavender solid waste was ground through a sieve of 4 mm diameter and a sieve of 1 mm diameter. These two samples, along with a sample of untreated solid waste, were extracted in the ultrasonic homogenizer under the same optimized conditions, with the aim to determine if the grinding degree can be considered as a key factor in the extraction process. As performance criteria for the process, the DPPH and FRAP assays and TPC measurement were used.

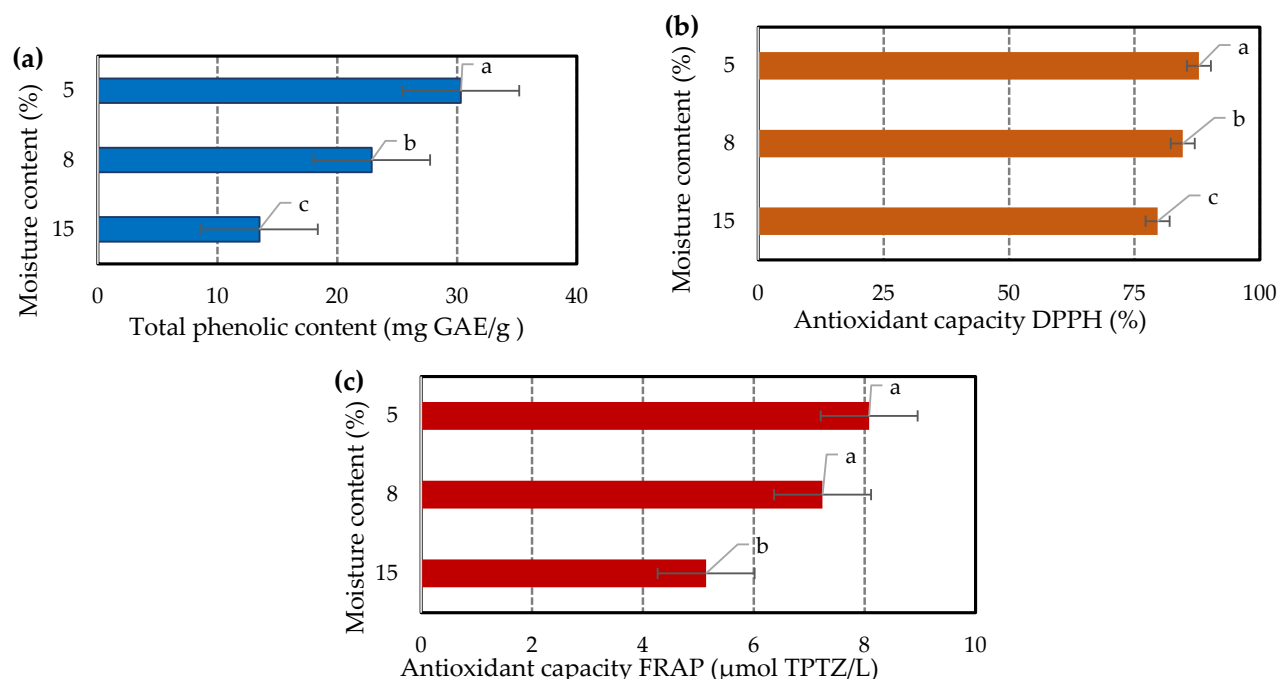


Figure 3. Effect of moisture content of lavender solid waste on (a) total phenolic content of extract (GAE mg/g of waste), (b) antioxidant capacity of extract (DPPH, %), and (c) antioxidant capacity (FRAP, μmoles TPTZ/L). The same letters denote no-statistically significant-difference-between-the-values in-the-same-column($p > 0.05$).

Upon examining the graphs (Figure 4), it is apparent that the 1 mm sieve-ground sample exhibits superior behavior. However, following statistical analysis, it was determined that variations in the milling degree do not result in significant changes (p -value > 0.05) in terms of the TPC and % antioxidant capacity. In the FRAP assay, a statistically significant increase (p -value < 0.05) was observed in the 1 mm sieve-ground sample compared to the unground one. Nevertheless, this finding is deemed insufficient to conclusively designate the milling degree as a key factor in maximizing the extraction efficiency.

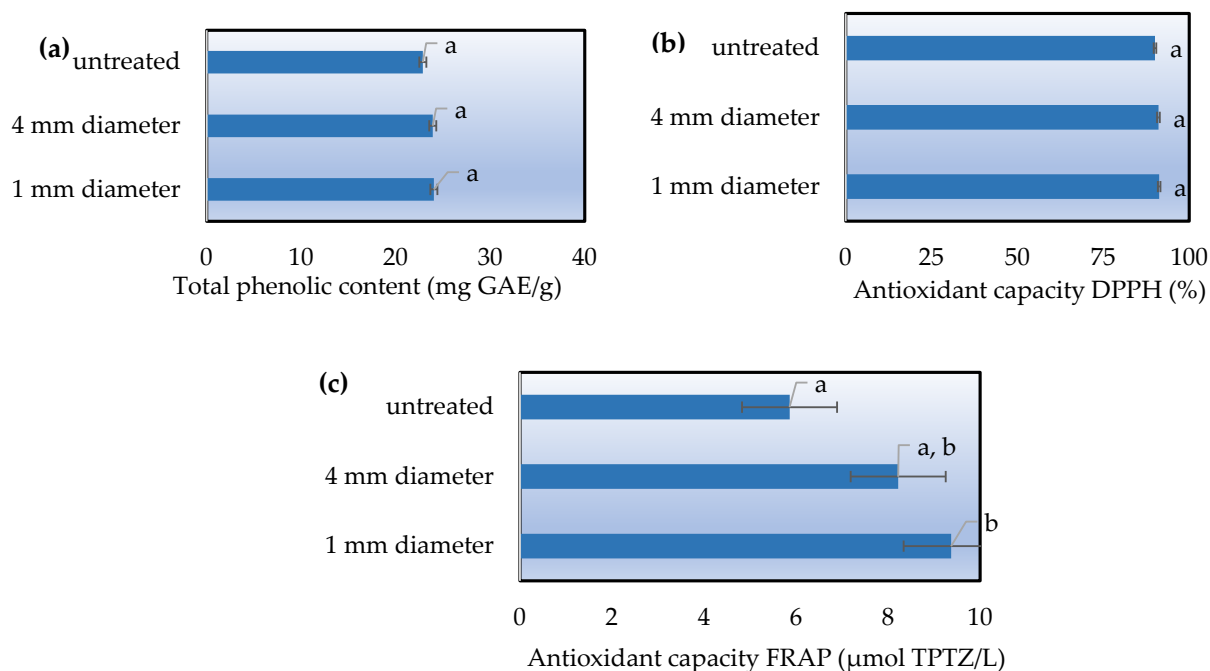


Figure 4. Effect of milling degree of lavender solid waste. (a) Total phenolic content of extract (GAE mg/g of waste), (b) antioxidant capacity of extract (DPPH, %), and (c) antioxidant capacity (FRAP, μmoles TPTZ/L). The same letters denote no-statistically significant-difference-between-the-values in-the-same-column($p > 0.05$).

3.3. Optimization of Microwave-Assisted Extraction of Lavender Solid Waste

In all experiments, the extraction yield (Y) was time dependent and increased with prolonged times, particularly from 1 to 4 min, but increased slowly from 4 to 30 min. Thus, the effective extraction time for attaining maximum yield was about 4 min. Two different solvents were studied, aqueous ethanol and aqueous methanol. The results for each type of solvent are presented in Figure 5.

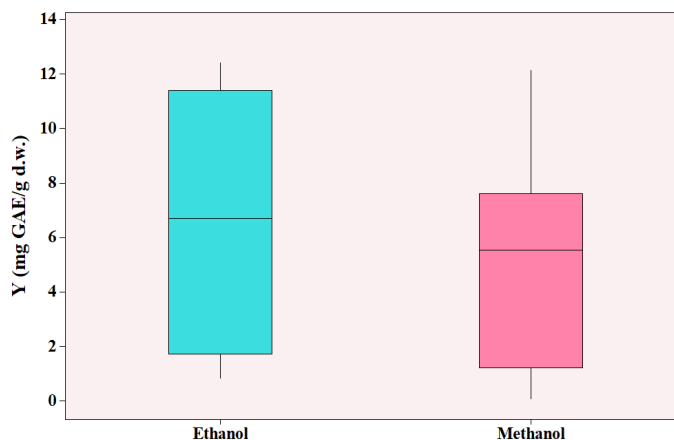


Figure 5. Box plot showing the effect of solvent type on extraction yield Y (mg GAE/g d.w.).

As shown in Figure 5, ethanol led to higher yields, which ranged from 0.82 to 12.4 mg GAE/g d.w. (dry weight), whereas methanol yielded 0.08–12.1 mg GAE/g d.w.; for both cases, the lowest value was observed when pure solvent was used (no water added).

It should be noted that methanol's highest TPC value was acquired in experimental conditions where methanol was not present (experiment 2 of Table 1) and the solvent was pure water. The highest yield with methanol present as solvent was 8.2 mg GAE/g d.w. (experiment 4 of Table 1). The effect of the solvent type was found to be statistically significant ($p = 0.004$). Furthermore, methanol is considered toxic for humans and can cause severe environmental problems [44]. Agustin-Salazar et al. [44] studied the effect of ethanol and methanol for the extraction of grape marc and reported that ethanol had a superior or equal effect on the total phenolic content, total flavonoid content, and antioxidant ability, while being environmentally friendly. As a result, ethanol was the solvent of choice for the current study of MAE.

Microwave-assisted extraction is based on the ionic conduction and dipole rotation that occurs when microwave power is given to any system able to absorb it. Different solvents have various dielectric constants and loss tangent factors that are related to their ability to transform electromagnetic radiation to heat, enhancing diffusion and solubilization phenomena [45,46]. Ethanol, methanol, and water have loss tangent values of 0.892, 0.522, and 0.123, respectively, at 25 °C [17]. Because of this, ethanol is more capable than methanol of absorbing microwaves and turning them into heat, accelerating the breakdown of plant material [17,47]. Ivanov et al. [15] also reported that ethanol led to better results regarding the ultrasound-assisted extraction of lavender solid waste compared to methanol. In addition, the higher efficiency of ethanol could be possibly attributed to its polarity and better affinity with the phenolic compounds of lavender flower, thus enabling better isolation from the plant matrix. The higher extraction efficiency of ethanol compared to methanol was also reported by Kaderides et al. [17] for the MAE of polyphenols from pomegranate peels, Amirabbasi et al. [48] for the phenolics extraction from okra stems, and Do et al. [49] for the isolation of phenolics and flavonoids from *Limnophila aromatica*.

Figure 6 presents the effect of microwave power (P), solvent/SW ratio (L/S), and solvent concentration (S) on extraction yield with ethanol as solvent. Microwave power is one of the most important factors in MAE, since it greatly facilitates the diffusion of the solvent into the plant tissue matrix and enhances mass transfer phenomena. It is also associated with the rate at which heat accumulates in the extraction system, which affects the rate of plant tissue deterioration along with the occurrence of chemical reactions. The accumulation of heat inside the cell also reduces liquid rarefaction, thus increasing the liquid mobility in the plant matrix and the penetration of solvent in the cells. In addition, it causes the evaporation of moisture, thus increasing the internal pressure on the cell wall. Finally, the pressure gradient with the outer environment of the cell escalates and the cell ruptures, causing the release of the active constituents [45–47]. The importance of power level was evident in the results, since lower power levels (100 W) had the lowest yields in TPC, whereas an increase in microwave power had a positive effect in achieving the highest TPC values. The yield actually diminished when the power was at 800 W; however, this could be attributed to the nature of these experiments, where extreme conditions were examined. The experiment at 800 W with pure water resulted in a relatively high yield (11.5 mg GAE/g d.w.), whereas the other experiment with pure ethanol had a really low yield (2 mg GAE/g d.w.). Pimentel-Moral et al. [50] reported that the higher temperatures (up to 164 °C) induced by microwaves had a positive effect on the MAE of hibiscus, and similar results were also found by Casazza et al. [51] for the MAE of apple peels. Considering that MAE in the current study was conducted only for 4 min, sufficiently high-power levels were mandatory to enable the rapid heating of the solvent. Kaderides et al. [17], for the MAE of pomegranate peels, and Solomakou et al. [50], for the MAE of spent coffee grounds, are also in agreement with the findings of the current study.

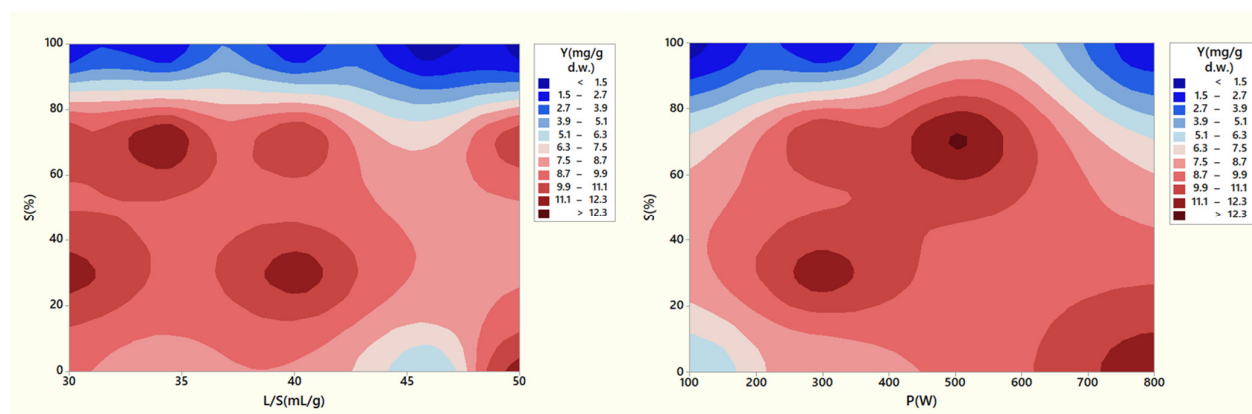


Figure 6. Effect of microwave power P (W), solvent/SW ratio L/S (mL/g), and solvent concentration S (%) on extraction yield Y (mg GAE/g d.w.).

Ethanol concentration in the solvent solution exhibited a significant effect on the extraction yield. Considering Figure 6, it was observed that whereas a water system had satisfactory results in yield values, the introduction of ethanol to the water had a positive effect. Ethanol and water have relative polarities of 0.654 and 1.0, respectively (Kaderides et al., 2019 [17]). This disparity in polarity has a beneficial effect in the extraction of phenolics, as a bipolar medium could potentially influence a wider variety of phenolic compounds. This principle is called “polarity versus polarity” and its basis is that the higher the molecular weight of the solvent, the lower the polarity, which allows other substances of about the same molecular weight to be easily extracted [51]. In addition, as previously mentioned, the presence of ethanol in the solution enables higher efficiency in conversion of microwave power to heat because of ethanol’s dielectric properties. Moreover, ethanol has the ability to effectively dissolve constituents of the cell wall and enable their dissolution and availability in the main solvent volume [52–54]. However, with an increasing ethanol concentration in the solution, there is an equilibrium up until negative effects start to occur, as was the case for high S values (above 70%). Chlev et al. [31] similarly reported that a 40% aqueous ethanol had the highest yield in extracting rosmarinic acid, caffeic acid, and luteolin from lavender distillation solid waste compared to 70% and, in particular, 100% ethanol solutions. This phenomenon may be ascribed to a diminished capacity for the transfer of extracted phenolic compounds from the cellular matrix to the bulk solution, primarily owing to the absence of water. This absence results in reduced solubility. Furthermore, the overheating is instigated by ethanol’s strong affinity for microwave energy, leading to the degradation of these thermosensitive compounds. Turrini et al. [16] also utilized a 70/30 ethanol/water system to facilitate the extraction of polyphenols from steam-distilled lavender solid waste. Similar findings were reported for the MAE of various waste streams such as grape by-products, spent coffee grounds, pomegranate peels, and apple peels [17,51,52,55,56].

In terms of the liquid-to-solid ratio (L/S), our observations indicate that it was the least important factor in the study. To ensure the effectiveness of our experimental conditions, we initially set a relatively high L/S concentration, 30 mL/g. Consequently, further adjustments to increase the L/S ratio did not yield any significant effects. The most favorable results were obtained within the approximate range of 30–40 mL/g. When the liquid volume exceeded a certain threshold, the available microwave energy proved insufficient to swiftly elevate the solvent’s temperature and expedite the extraction process. Additionally, this excess liquid could result in over-dilution of the extracts, leading to increased processing costs [52,57–59]. Notably, the liquid-to-solid ratio was the only factor that was found to be statistically insignificant (p -value > 0.05).

The statistical analysis of variance was performed for the ethanol microwave-assisted extraction, and the results are presented in Table 2.

Table 2. Analysis of variance for the MAE of phenolic compounds from lavender solid waste.

Source	DF	F-Value	p-Value
Model	9	59.23	0.000
Linear	3	40.13	0.000
P (W)	1	20.97	0.004
S (%)	1	27.50	0.002
L/S (mL/g)	1	0.33	0.586
Square	3	80.86	0.000
P (W) * P (W)	1	3.04	0.132
S (%) * S (%)	1	74.44	0.000
L/S (mL/g) * L/S (mL/g)	1	0.39	0.557
2-way interaction	3	4.22	0.063
P (W) * S (%)	1	3.73	0.102
P (W) * L/S (mL/g)	1	0.74	0.422
S (%) * L/S (mL/g)	1	1.47	0.271
Lack of fit	5	30.79	0.136
MODEL SUMMARY			
S	R ²	R ² adjusted	
0.805	98.89%	97.22%	

A coefficient of variability (R^2) of 98.89% indicates that the variability of the response was well captured by the proposed model and the experimental data. Furthermore, the adjusted R^2 at 97.22% suggests a high level of agreement between the experimental and predicted yield values. The model's F -value, which stands at 59.23 with a p -value of 0.000, underscores its significance. Additionally, the lack-of-fit test, with a non-significant p -value (p -value > 0.05), confirms that the regression model is crucial and aligns well with the experimental data. Specifically, both the power (P) and solvent concentration (S) were found to be statistically significant (p -value < 0.05). However, there were no detected interactions between the model's parameters.

The desirability profile for optimum yield showed that the highest desirability level (on a scale of 0–1) can be achieved with a power of 800 W, a solvent/solid ratio of 30 mL/g, and 34% aqueous ethanol as solvent for an extraction time of 4 min (Figure 7). Under these conditions, the predicted extraction yield was 16.487 mg GAE/g d.w., whereas the observed experimental value was 14.7 ± 0.3 mg GAE/g d.w.

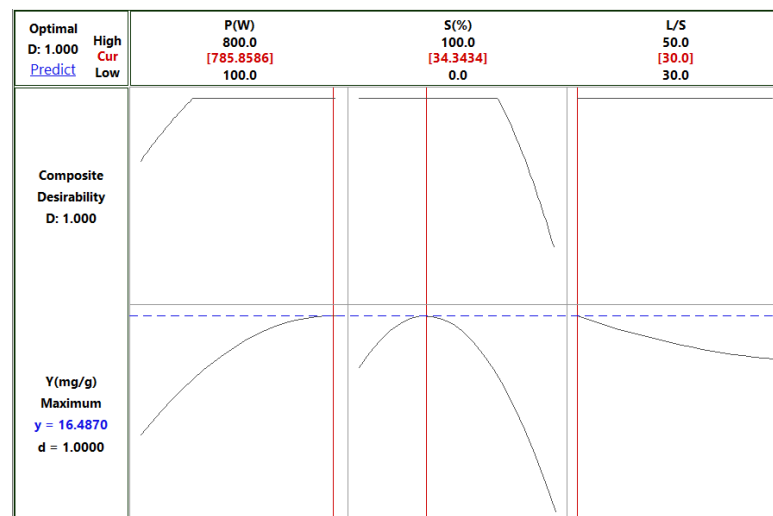


Figure 7. Optimization for the MAE of phenolic compounds from lavender solid waste.

3.4. Drying of Lavender Solid Waste Extract

Spray-drying the UAE extract had a 20% solids yield without an encapsulation agent; however, this increased to 52% when 10% w/v maltodextrin (10 DE) was added to the extract prior to drying. Comparatively, the yield for the MAE extract was 40% without maltodextrin, rising to 55% with the encapsulation agent. The notable disparity in the non-encapsulated extracts could be attributed to intrinsic variations in the physical and chemical properties of the extracts. It is also noteworthy that the MAE extract without maltodextrin exhibited lower stickiness on the walls of the drying chamber, suggesting that this could potentially contribute to the observed variations in yield. Although the obtained yield using maltodextrin is acceptable [60], it is strongly suggested that some key parameters (inlet temperature, maltodextrin concentration, etc.) should be optimized so that the process can be possibly applied on an industrial scale. The yields obtained from freeze-drying ranged between 70 and 75%. The higher efficiency of the freeze-drying method is also noted by Kandasamy et al. [61] in their review regarding the efficiency of the two encapsulation techniques, wherein freeze-drying is also demonstrated as a more costly and time-consuming method.

A primary objective of the experiment was to investigate whether various extraction and drying methods adversely affect the antioxidant properties and polyphenolic content of the extract. Focusing on ultrasound-assisted extraction (UAE) and microwave-assisted extraction (MAE) techniques, alongside freeze-drying and spray-drying processes, this research also contemplates the impact of maltodextrin as an encapsulation medium. The detailed findings are presented in Table 3, with a particular focus on assessing to what extent the resulting powders retain the same polyphenolic content and antioxidant activity as the original extracts.

Comparing the effects of drying techniques on polyphenolic content, freeze-drying emerges as the superior method for preserving total phenolic content (TPC), as measured using the Folin–Ciocalteu method. Notably, freeze-drying ensures the retention of higher TPC levels both with and without the addition of maltodextrin in comparison with the spray-drying method (Table 3). Specifically, during freeze-drying, a statistically insignificant degradation of phenolic compounds is noted (p -value > 0.05), whereas in the case of spray-drying, despite maltodextrin supplementation, a statistically significant degradation is observed (p -value < 0.05). Additionally, ultrasound-assisted extraction (UAE) seems to outperform microwave-assisted extraction (MAE), both on the extract and in the powder form. Maltodextrin, employed as an encapsulation agent, elicits varied effects on the TPC. In the context of UAE extracts, the addition of maltodextrin contributes to enhanced TPC preservation. Conversely, for MAE extracts, the utilization of maltodextrin leads to a small reduction (p -value > 0.05) in TPC compared with the extract without maltodextrin during the drying process. The different behaviors of UAE and MAE extracts on the encapsulation medium can possibly be attributed to the significant difference in the yield of the spray-drying process on the non-encapsulated extract (20% for the UAE versus 40% for the MAE extract). Based on this, it is safe to infer that maltodextrin could act supportively on the MAE extract if added in a smaller amount. In summary, the influence of maltodextrin on the TPC is contingent upon the extraction method employed, underscoring its varied effects on polyphenolic preservation in distinct extraction and drying processes.

The preservation of antioxidant capacity, as assessed through the FRAP and DPPH radical scavenging assays, appears to be better enhanced in the freeze-drying method in comparison with the spray-drying method, aligning with the trends observed for the total phenolic content. It is also observed that the UAE method consistently exhibits higher FRAP and DPPH values than the MAE method (Table 3). This trend also aligns with the earlier observation that UAE tends to outperform MAE in preserving the TPC. The addition of maltodextrin appears to have a non-significant impact on the FRAP and DPPH values. Whereas the non-encapsulated UAE spray-dried extract shows slightly higher

FRAP and DPPH values (p -value > 0.05) than its maltodextrin-supplemented counterpart, the opposite trend is observed in the MAE spray-dried extracts.

In conclusion, the comprehensive evaluation employing the FRAP, DPPH, and Folin–Ciocalteu methods consistently points to the freeze-drying method, particularly when accompanied by maltodextrin, as the optimal approach for preserving the antioxidant capacity of lavender extracts. Similar results regarding the better quality of the freeze-dried extracts in terms of TPC and the DPPH and FRAP assays have been obtained by several researchers [62–65] and are based on the dehydration of a frozen product: due to the absence of liquid water and the low temperatures required for the process.

It is also concluded that the UAE method exhibits higher values in terms of the TPC and the FRAP and DPPH assays. Previous experimental findings have also indicated that UAE is more effective in extracting total phenolics from lime peel waste with high antioxidant activity, saving 33% of time compared to MAE [66]. Espada-Bellido et al. [67] noted that non-significant differences were obtained when using UAE and MAE at their respective optimum conditions for the extraction of TA and TPC from blackberry. In contrast, Dahmoune et al. [68] reported that the MAE extract of *Myrtus communis* L. leaves was the best in terms of antioxidant activity and total phenolic content compared to UAE extracts. In addition, they reported that the antioxidant activity of the extract depends on the process as well as on the plant type and the assay applied. From another perspective, Shen et al. [69] demonstrated that the combination of ultrasonic-assisted extraction and microwave-assisted extraction (UMAE) has been shown to be a highly efficient and cost-effective extraction technique.

Table 3. Effect of drying and encapsulation on the extract’s antioxidant properties.

Sample	Total Phenolic Content (mg GAE/g Dry Lavender)	% Antioxidant Capacity	FRAP ($\mu\text{mol TPTZ/L}$)
UAE extract	21 $\pm$ 4 a	89 $\pm$ 1 a	12 $\pm$ 1 a
MAE extract	14.7 $\pm$ 0.3 b	72 $\pm$ 1 b	7 $\pm$ 1 b, c
Spray-dried UAE extract	15 $\pm$ 1 b, c	72 $\pm$ 1 b	7.7 $\pm$ 0.8 b
Spray-dried UAE extract with 10% maltodextrin	17 $\pm$ 2 c	84 $\pm$ 3 c	9 $\pm$ 1 a, b
Spray-dried MAE extract	12 $\pm$ 1 d	91.0 $\pm$ 0.3 d	5.58 $\pm$ 0.08 c
Spray-dried MAE extract with 10% maltodextrin	10 $\pm$ 1 d	87.0 $\pm$ 0.9 e	5.6 $\pm$ 0.4 c
Freeze-dried UAE extract	16 $\pm$ 2 b	75 $\pm$ 1 f	9 $\pm$ 3 a
Freeze-dried UAE extract with 10% maltodextrin	20.4 $\pm$ 0.9 a	86.3 $\pm$ 0.2 e	11 $\pm$ 1 a

The same letters denote no statistically significant difference between the values in the same column (p > 0.05).

Moisture content

The moisture content for all the obtained dry extracts ranged between 3.1 and 6.0% (Table 4). These values were in the desired range of quality parameters for a powder to be considered a dry product [70]. The values of the freeze-dried powders were found to be lower than these of their spray-dried counterparts. Similar results were also obtained by Lee et al. [62] and Jovanovic et al. [71] in their research and were attributed to the rapid spray-drying process that caused the dried powder to be developed into a complete amorphous state that tends to absorb a high amount of moisture during the powder handling process after spray-drying and to the higher transition temperature of spray-dried samples. Accordingly, slightly increased values were observed in the obtained powders due to the addition of maltodextrin. A similar trend has been reported by Goula and Adamopoulos [72] and can be attributed to the fact that it is difficult for water molecules to diffuse past the larger maltodextrin molecules.

Bulk density

The bulk density of the dry extracts is an important property when considering the transportation, storage, packaging, and mixing processes. The bulk densities of the lyophilized and sprayed-dried powders ranged between 320 and 385 and 300 and 456 mg/mL, respectively, with slightly higher values for the spray-dried samples compared to the corresponding lyophilized samples (Table 4). The MAE extract was found to have significantly higher bulk density. It was noted that the bulk density increased with the addition of maltodextrin as a drying agent. This phenomenon could be explained by agglomerate formation in the presence of maltodextrin due to a delay in the formation of a semi-permeable layer by the internal components during drying [70].

Hygroscopicity

The obtained sprayed-dried extracts showed a moderate hygroscopicity of between 8.3 g and 16.9 g per 100 g of dry solids (Table 4). The lowest values were observed when maltodextrin was used. This is due to the fact that maltodextrin is a material with low hygroscopicity and confirms its efficiency as a carrier agent [73]. Moreover, the powders obtained using the extract from ultrasonic extraction as the feed presented slightly lower values for hygroscopicity in comparison with the values from microwave extraction. In general, the observed values are in accordance with the existing literature on medical plant extracts [70,74,75]. The freeze-dried extracts were observed to have the lowest hygroscopicity values (Table 4).

Water activity

Water activity is a determining factor that influences microorganism growth and various chemical/biochemical reactions within the matrix and, as a result, the shelf life and quality of the product. It can be observed in Table 4 that all powders showed a water activity of below 0.6, which is considered microbiologically stable [76]. The water activity values were affected significantly (p -value < 0.05) by the drying technique and the addition of maltodextrin. Specifically, spray-dried extract powders with maltodextrin showed the lowest a_w values (Table 4). These results are consistent with other studies [77,78].

Table 4. Comparison of physicochemical properties in the powder samples.

Sample	Moisture Content (% Wet Basis)	Bulk Density (mg/mL)	Hygroscopicity (g Water/100 g Powder)	Water Activity (a_w)
Spray-dried UAE extract	4.4 ± 0.6	300	13.0 ± 0.7 a	0.42 ± 0.01 a
Spray-dried UAE extract with 10% maltodextrin	4.6 ± 0.3	363	9.7 ± 0.9 b	0.33 ± 0.02 b
Spray-dried MAE extract	5.1 ± 0.5	350	16.9 ± 0.4 c	0.53 ± 0.04 c
Spray-dried MAE extract with 10% maltodextrin	6 ± 1	456	15.04 ± 0.09 d	0.48 ± 0.04 d
Freeze-dried UAE extract	3.1 ± 0.8	320	11 ± 1 a, e	0.55 ± 0.08 c
Freeze-dried UAE extract with 10% maltodextrin	3.2 ± 0.7	385	8.3 ± 0.8 b, e	0.48 ± 0.07 d

There are no statistically significant differences between the values in the same column with the same letter. ($p > 0.05$).

4. Conclusions

This study aimed to investigate the impact of various parameters on the extraction efficiency of lavender solid waste, including moisture content and grinding degree. The drying curve analysis revealed distinct phases in the drying process, emphasizing the importance of monitoring and controlling moisture levels to prevent product degradation. The comparison of samples with different moisture contents demonstrated a significant

influence on antioxidant activity, with the 5% moisture content sample exhibiting superior results.

Additionally, the grinding degree of lavender solid waste was explored, with ultrasound extraction conducted under controlled conditions. Whereas the 1 mm sieve-ground sample displayed superior behavior, statistical analysis indicated that variations in the milling degree did not lead to significant changes in the total phenolic content and antioxidant capacity. Therefore, the role of grinding degree in maximizing the extraction efficiency remained inconclusive.

Furthermore, the study delved into the optimization of the microwave-assisted extraction (MAE) of lavender solid waste. Ethanol was identified as the preferred solvent due to its higher efficiency in extracting phenolic compounds compared to methanol. The optimization process revealed that a power of 800 W, a solvent/solid ratio of 30 mL/g, and 34% aqueous ethanol as solvent for 4 min yielded the highest desirability level and an observed extraction yield of 14.750 ± 0.350 mg GAE/g d.w.; UAE seemed to outperform the MAE method in terms of the TPC and the DPPH and FRAP assays.

Subsequent investigations into the drying of lavender solid waste extracts using different methods (spray-drying, freeze-drying) and the use of maltodextrin as a drying agent demonstrated varying impacts on the antioxidant properties and polyphenolic content. Freeze-drying, particularly when accompanied by maltodextrin, was identified as the optimal approach for preserving the antioxidant capacity of lavender extracts. Additionally, this study highlighted the influence of drying techniques and encapsulation using maltodextrin on the physicochemical properties, such as moisture content, bulk density, hygroscopicity, and water activity.

In summary, this research provided valuable insights into optimizing the extraction and drying processes for lavender solid waste, contributing to the understanding of the factors influencing the quality and efficiency of lavender extract production from distillation wastes. These findings have implications for industries utilizing lavender and possibly other aromatic/medicinal plant extracts in various applications, including pharmaceuticals, cosmetics, and food products.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/separations11030067/s1>. Figure S1: Main effects plots for the effect of extraction time and ultrasound amplitude on (a) total phenolic content of extract (GAE mg/g of waste), (b) antioxidant capacity of extract (DPPH, %), and (c) antioxidant capacity (FRAP, μ moles TPTZ/L); Figure S2. Optimization for the UAE of phenolic compounds from lavender solid waste for ethanolic and methanolic solvent; Table S1: Experimental design for the UAE of lavender solid waste for two different solvents.

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