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The Effect of Spray-Drying Conditions on the Characteristics of Powdered *Pistacia lentiscus* Leaf Extract

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Abstract: *Pistacia lentiscus* is an evergreen shrub widely used in folk medicine due to the high biological potential of the leaves' phenolic compounds. Since phenolic compounds are susceptible to degradation under different heat, light and oxygen conditions, various microencapsulation techniques, such as spray drying, can be used to increase their stability. The objective of this study was to examine the influence of different carriers (gum arabic (GA), maltodextrin 13–17 DE (MD), β-cyclodextrin (BCD) and their mixtures), carrier concentrations in feed (12.8, 16 and 19.2% (*m/V*)) and drying temperatures (120, 150 and 180 °C) on the physiochemical properties, total phenolic content (TPC) and antioxidant activity (AOA) of *Pistacia lentiscus* leaf extract powders. Product yields of powders ranged from 23.53 to 65.51%, moisture content from 2.89 to 12.03%, hygroscopicity up to 4.45 g/100 g, solubility from 27.11 to 86.84% and bulk density from 0.24 to 0.45 g/mL. All obtained powders had satisfactory physicochemical properties, except BCD powders, which resulted in the lowest product yield, solubility and bulk density. However, BCD powders and those produced with GA containing mixtures retained the highest amounts of TPC and AOA. Thereby, the carrier mixtures with GA at a concentration of 19.2% and dried at 150 °C are recommended as the most suitable for the production of encapsulated *Pistacia lentiscus* leaf extracts with desirable physicochemical properties, rich in phenolics and with high antioxidant activity.

Keywords: *Pistacia lentiscus*; spray drying; carrier type; phenolic content; antioxidant activity

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1. Introduction

Pistacia lentiscus L. (mastic tree) is an evergreen shrub that grows in the Mediterranean and Middle East. It is well adapted to harsh growing conditions, drought and warm environments. In folk medicine, the plant is used for jaundice; gastrointestinal, kidney and liver diseases; and the treatment of hypertension, diabetes, heart disease, cough, sore throat, eczema, etc. [1]. Extracts from *P. lentiscus* leaves are rich in phenolic compounds, especially tannins and flavonoids [2], and exhibit antioxidant [3–5], anti-inflammatory [6], blood-sugar-lowering [7], antimicrobial [8] and anthelmintic [9] activities, which makes them interesting for human health as dietary supplements. Plant-derived phenolic compounds have low stability to pH, enzymes, temperature, oxygen and light [10]. Due to the presence of unsaturated bonds in their structures, phenolic compounds are susceptible to oxidation and need to be encapsulated to increase their stability to environmental factors [11]. Microencapsulation is a process in which tiny particles or droplets are surrounded by a coating or embedded in a homogeneous or heterogeneous matrix to form small capsules. Many techniques have been developed for the microencapsulation of food ingredients, but spray drying is the most commonly used technology in the food industry due to its low cost and available equipment [12]. The properties of the produced spray-dried powders depend on the drying temperature, drying air flow rate, feed flow rate, atomizer, grade

and the concentration of the carrier [13,14]. The most commonly used carriers are gum arabic (GA) and maltodextrin (MD). GA has high solubility, low viscosity [15] and good emulsifying properties [16] but is expensive and contains impurities [15]. Maltodextrins, obtained by starch hydrolysis, are, on the other hand, cheap, have lower viscosity at higher concentrations and are odorless and almost tasteless [15,16]. Another type of carrier is β-cyclodextrin (BCD), a cyclic oligosaccharide with a nonpolar cavity and a hydrophilic exterior, which allows the formation of inclusion complexes with nonpolar guest molecules. Complexation improves the physiochemical properties of the entrapped molecules: it can improve their solubility; stabilize them against UV and visible light, heat and oxidation; control their volatility; and mask flavors and unpleasant odors by sublimation [17].

Several reports have been published on the improved characteristics of different plant extracts by spray-drying encapsulation—for instance, better physical properties in dandelion leaf extracts [18], improved physicochemical parameters in stevia leaf extracts [19] and remarkably improved storage stability in green tea leaf extracts [20]. However, to the best of our knowledge, there is no study of *P. lentiscus* leaf extract subjected to spray-drying microencapsulation. Therefore, the aim of this study was to investigate the spray-drying process of *P. lentiscus* leaf extract in terms of carrier type (GA, MD 13–17 dextrose equivalent (DE) and BCD, as well as a combination of GA with two other carriers), the concentration of carrier in the feed solution and the drying temperature, and to determine the influence of these parameters on the physiochemical properties, phenolic content and antioxidant capacity of *P. lentiscus* leaf extract powders.

2. Materials and Methods

2.1. Chemicals and Reagents

Maltodextrin DE 13.0–17.0 was purchased from Sigma-Aldrich (St. Louis, MI, USA); gum arabic, β-cyclodextrin, 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox), 98% 2,2′-azobis(2-methylpropionamidine) dihydrochloride, 98+% and dibasic sodium phosphate, 99+% were from Acros Organics (Acrõs Organics, Thermo Fisher Scientific, Geel, Belgium); 96% ethyl alcohol was purchased from Lach-ner (Neratovice, Czech Republic); 99.8% methanol and flourescein sodium salt was from Honeywell Research Chemicals (Bucharest, Romania) and purified/diluted water was prepared in a Milli-Q water purification system (Millipore Corp., Bedford, MA, USA). Anhydrous sodium carbonate was from Gram-Mol (Zagreb, Croatia); Folin–Ciocalteu reagent (FC) was from Kemika (Zagreb, Croatia) and sodium hydroxide pellets were from the Carlo Erba Reagents GmbH (Emmendingen, Germany).

2.2. Plant Material

Samples of *P. lentiscus* L. leaves were collected on the island of Korčula, Croatia (coordinates 42.961182 N, 16.721574 E) and botanically identified with the support of the Faculty of Agriculture, University of Zagreb (Zagreb, Croatia). The samples were collected in January 2021, dried at room temperature in the shade to a constant mass and packed in paper bags. They were ground in a commercial grinder (GT11, Tefal, Rumilly, France) prior to extraction.

2.3. Preparation of the P. lentiscus Leaf Extracts

Approximately 20 g of dried *P. lentiscus* leaves were ground and placed in a jar. A volume of 500 mL of 70% ethanol was added. The mixture was placed in an ultrasonic bath S 40H (Elmasonic, Elma, Singen, Germany) at 50 °C for 30 min. The extract was then filtered through a filter paper into a collection vessel and stored at −20 °C until further experiments were performed.

2.4. Spray Drying

Powders from the extracts of *P. lentiscus* leaves were prepared according to a full factorial experimental design on a laboratory-scale spray dryer SD 06 (Labplant, North Yorkshire, UK). MD and GA are common wall-forming materials [15], and, as mentioned

above, BCD forms complexes with aromatics and thereby improves the physiochemical properties of the entrapped molecules [17]. MD 13–17 DE, GA and BCD, as well as a mixture of MD 13–17 and GA and a mixture of BCD and GA, were dissolved in purified water and 100 mL was added to 100 mL of the prepared extract to prepare the feeding solution with carrier concentrations of 12.8, 16 and 19.2%, which was spray-dried at three inlet drying temperatures (120, 150 and 180 °C). The carrier concentration range was selected based upon the research of Pudziuvelyte et al. [21], who used similar carriers in a concentration range from 10% to 30% in the feeding solution.

The feed solution was stirred at room temperature on a magnetic stirrer (HSC Ceramic Hot Top-Plate Stirrer, VELP Scientifica Srl, (Usmate Velate MB, Italy) for approximately 30 min prior to the spray-drying process. The corresponding outlet temperatures were 58–77, 72–91 and 76–100 °C. All powders were prepared in duplicate and placed in plastic containers, blown out with nitrogen, sealed with parafilm and stored at −20 °C until further analysis.

2.5. Methods of Analysis

2.5.1. Product Yield

The product yield was calculated as the ratio between the mass of powder produced and the dry matter content of the initial feed solution according to Equation (1) [21].

$$\text{Product yield } (\%) = \frac{Mass\ of\ the\ powder\ obtained\ at\ the\ spray\ dryer}{Solid\ content\ of\ the\ inital\ feed\ solution} \times 100 \quad (1)$$

2.5.2. Moisture Content

To determine the moisture content (%), the powders were dried in an oven at 105 °C (FN 500; Nueve, Ankara, Turkey) until a constant mass was obtained. The moisture content was calculated as the difference in the mass before and after drying [22] (AOAC).

2.5.3. Hygroscopicity

Hygroscopicity measurements were performed according to the method described by Šavikin et al. [23] with slight modifications. A mass of 1 g of each powder was placed in a desiccator containing saturated NaCl solution (RH = 75.3%) at room temperature. After one week, the powders were weighed and hygroscopicity was expressed in g of adsorbed water per 100 g of powder using the following equation:

$$\text{Hygroscopicity } (\text{g}/100\text{g}) = \frac{m_7 - m_0}{m_0} \times 100 \quad (2)$$

where m_0 is the mass (g) of the powder before storage and m_7 is the mass (g) of the powder after 7 days of storage.

2.5.4. Solubility

The solubility of the powders was measured according to the modified method described by Anderson et al. [24]. A mass of 1 g of powder was dissolved in 10 mL of distilled water in glass tube. The mixture was stirred for 1 min with a vortex vibrator (Vorteks Velp Scientifica ZX3, Usmate Velate MB, Italy). It was then thermostatted at 37 °C for 30 min in a water bath (B-490; Büchi, Flawil, Switzerland), followed by centrifugation at 5500× g for 20 min (Rotofix 32; Hettich, Tuttlingen, Germany). The supernatant obtained was dried in a laboratory oven at 105 °C (FN 500; Nueve, Turkey) until a constant mass. The solubility was calculated as follows:

$$\text{Solubility} = \frac{m(s)}{m(p)} \quad (3)$$

where *m(s)* is the mass (g) obtained by drying the supernatant and *m(p)* is the mass (g) of the powder analyzed.

2.5.5. Bulk Density

The bulk density (g/mL) was determined by placing 1 g of powder in a 10 mL graduated cylinder and subjecting the cylinder to vibration for 1 min. The ratio between the powder mass and the volume occupied in the cylinder determined the bulk density value [25].

2.5.6. Extraction of the Phenolic Compounds from the Powders

The phenolic compounds were extracted from the powders with 80% methanol. A mass of 1 g of powder was weighed into a test tube and 10 mL of 80% methanol was added. The extraction of the samples was carried out ultrasonically assisted at a temperature of 50 °C for 20 min. Subsequently, the extracts were filtered through a 0.22 µm OF-NY-17022 microfilter into vials and stored at −60 °C until further use. The samples were used for the determination of total phenolic content and antioxidant activity.

2.5.7. Total Phenolic Content (TPC)

Total phenolic content (TPC) was measured using the FC reagent. First, 100 µL of extract, 200 µL FC reagent and 2 mL distilled water were added to a glass tube. After 3 min, 1 mL of concentrated sodium carbonate solution (20% w/v) was added and the samples were incubated in a water bath at 50 °C for 25 min. Subsequently, absorbance was measured at 765 nm (Uviline 9400 UV-Vis spectrophotometer, Secomam, Champigny sur Marne, France). The results were expressed as mg of total phenolic content per g of dry matter of the extract in gallic acid equivalents (GAE) [26].

2.5.8. Antioxidant Activity (AOA)

The antioxidant activity of *P. lentiscus* leaf encapsulated extracts was determined by the oxygen radical absorption capacity (ORAC) assay (CLARIOstar Microplate Reader, BMG LABTECH, Ortenberg Germany). Solutions of fluorescin (70.3 nM), 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) (0.13185 mol/L) and 2, 2′-azobis(2-amidino-propane) dihydrochloride (AAPH) (240 mM) were prepared in phosphate buffer (75 mM, pH = 7.5). Serial dilutions of Trolox solution were used to generate the standard curve. Then, 25 µL appropriately diluted samples, serial dilutions of Trolox and the blank solution (phosphate buffer) were added to a 96-well black plate containing a fluorescein solution (150 µL). The microplate was incubated for 30 min at 37 °C, and after three cycles (representing the baseline signal), freshly prepared AAPH (25 µL) was injected through a software-controlled injector. Fluorescence intensity was measured every 90 s (λ eks. 485 nm i λ em. 528 nm) and lasted up to 120 min. The collected data were analyzed using the MARS software (MARS 2.0 software, BMG LABTECH, Ortenberg, Germany) [3].

2.6. Statistical Evaluation

Experimental design and statistical data processing were performed using the Statistica 12.0 software (StatSoft, Inc., Tulsa, OK, USA). The experiments were designed as a mixed full factorial experimental design with one factor at five levels and two factors at three levels. The influences of the carrier type (BCD, MD 13–17, GA, MD + GA and BCD + GA), carrier concentration in the feed solution (12.8%, 16% and 19.2%) and temperature (120, 150 and 180 °C) were considered as independent variables, resulting in a total of 45 experimental runs, performed in duplicate. The responses obtained from the experimental design were the physicochemical parameters, product yield (%), moisture content (%), hygroscopicity (g/100 g), solubility (%), bulk density (g/L), total phenols (mg/g DM) and antioxidant activity (µmoL Trolox equivalent/g DM). All experiments and analyses were performed in duplicate. The normality and homoscedasticity of the data were analyzed using the Shapiro–Wilk test and Levene's test, respectively. The results obtained were analyzed by analysis of variance (ANOVA) and marginal means were compared using Tukey's HSD test or the Kruskal–Wallis test where appropriate. A statistically significant difference was considered at the level of $p \leq 0.05$ (95% confidence interval).

3. Results

In order to preserve the phenolic compounds and to achieve optimal physicochemical properties in the obtained powders to facilitate further processing and improve the storage stability, the process of the extracts' spray drying had to be optimized. In this study, the influence of three parameters (type and different ratios of carrier material to extract dry matter and temperature) on the product yield, moisture content, hygroscopicity, solubility, bulk density, total phenols and antioxidant activity of Pistacia lentiscus leaf extract powders was examined. The results and experimental design of the physical properties of the obtained *Pistacia lentiscus* leaf extract powders are shown in Table 1, and the results of the statistical analysis are summarized in Table 2. Table 3 shows the results and experimental design for the total phenolic content and antioxidant activity of the obtained *Pistacia lentiscus* leaf extract powders, while the statistical analysis is summarized in Table 4.

Table 1. Physical properties of the obtained *Pistacia lentiscus* leaf extract powders.

Carrier	m(Carrier):V(Extract)	Temperature	Product Yield	Moisture Content	Hygroscopicity	Solubility	Bulk Density
	(%)	(°C)	(%)	(%)	(g/100 g)	(%)	(g/mL)
BCD	12.8	120	29.17 ± 0.55	12.03 ± 0.03	1.98 ± 0.03	35.22 ± 0.87	0.35 ± 0.01
		150	43.06 ± 0.35	7.83 ± 0.04	1.22 ± 0.03	32.80 ± 0.79	0.32 ± 0.01
		180	49,12 ± 0.19	7.38 ± 0.02	3.08 ± 0.25	43.96 ± 0.59	0.24 ± 0.01
	16	120	38.33 ± 0.15	10.89 ± 0.02	2.12 ± 0.11	27.11 ± 0.60	0.32 ± 0.01
		150	40.27 ± 0.33	12.01 ± 0.03	1.63 ± 0.16	28.75 ± 0.56	0.35 ± 0.00
		180	42.68 ± 0.55	9.84 ± 0.03	4.45 ± 0.19	37.60 ± 0.59	0.34 ± 0.01
	19.2	120	33.56 ± 0.58	12.03 ± 0.01	1.78 ± 0.18	40.59 ± 1.33	0.30 ± 0.01
		150	27.42 ± 0.52	11.94 ± 0.01	0.45 ± 0.12	43.35 ± 0.45	0.24 ± 0.01
		180	23.53 ± 0.09	8.40 ± 0.03	3.60 ± 0.34	44.58 ± 0.41	0.25 ± 0.01
MD	12.8	120	47.91 ± 0.25	4.43 ± 0.03	0.08 ± 0.03	85.89 ± 0.83	0.39 ± 0.02
		150	49.96 ± 0.31	4.02 ± 0.02	0.26 ± 0.09	84.35 ± 0.55	0.38 ± 0.01
		180	48.13 ± 0.50	4.03 ± 0.03	0.07 ± 0.03	83.79 ± 0.43	0.33 ± 0.01
	16	120	53.61 ± 0.40	5.19 ± 0.02	0.06 ± 0.01	79.98 ± 0.69	0.45 ± 0.01
		150	52.44 ± 1.27	4.94 ± 0.04	0.32 ± 0.41	83.42 ± 0.18	0.38 ± 0.01
		180	55.47 ± 0.36	2.89 ± 0.02	0.12 ± 0.03	81.62 ± 0.36	0.40 ± 0.02
	19.2	120	62.69 ± 0.45	4.27 ± 0.02	0.08 ± 0.04	79.63 ± 0.39	0.45 ± 0.01
		150	61.25 ± 0.27	3.48 ± 0.02	0.16 ± 0.04	80.50 ± 0.14	0.39 ± 0.01
		180	62.81 ± 0.61	4.61 ± 0.02	0.07 ± 0.02	86.84 ± 5.44	0.35 ± 0.01
GA	12.8	120	55.16 ± 0.81	6.25 ± 0.02	2.23 ± 0.13	76.25 ± 0.49	0.31 ± 0.01
		150	51.92 ± 0.36	4.87 ± 0.02	3.95 ± 0.24	65.96 ± 0.86	0.33± 0.02
		180	52.95 ± 0.56	3.62 ± 0.02	4.03 ± 0.54	79.30 ± 0.50	0.39 ± 0.01
	16	120	60.49 ± 0.26	6.85 ± 0.02	2.50 ± 0.24	62.73 ± 0.64	0.31 ± 0.01
		150	40.57 ± 0.53	5.57 ± 0.02	3.37 ± 0.40	53.30 ± 0.40	0.29 ± 0.01
		180	38.08 ± 0.43	3.17 ± 0.01	3.93 ± 0.57	75.96 ± 0.86	0.35 ± 0.01
	19.2	120	54.15 ± 0.72	6.50 ± 0.02	2.56 ± 0.28	69.20 ± 0.45	0.34 ± 0.01
		150	62.43 ± 0.57	6.75 ± 0.04	1.54 ± 0.30	72.59 ± 0.62	0.29 ± 0.01
		180	59.84 ± 0.29	4.53 ± 0.02	3.40 ± 0.23	80.39 ± 0.42	0.31 ± 0.01
BCD + GA	12.8	120	42.10 ± 0.21	7.13 ± 0.03	1.40 ± 0.16	74.04 ± 0.58	0.33 ± 0.01
		150	60.96 ± 0.65	4.41 ± 0.01	2.69 ± 0.26	56.89 ± 0.76	0.30 ± 0.01
		180	59.09 ± 0.32	5.33 ± 0.02	4.40 ±0.28	73.07 ± 0.40	0.26 ± 0.01
	16	120	61.28 ± 0.39	6.23 ± 0.02	2.92 ± 0.43	67.96 ± 0.35	0.34 ± 0.01
		150	59.83 ± 0.44	3.91 ± 0.03	3.45 ± 0.40	75.74 ± 0.59	0.32 ± 0.01
		180	55.98 ± 0.11	4.68 ± 0.03	2.98 ± 0.38	54.79 ± 0.46	0.29 ± 0.01
	19.2	120	48.81 ± 0.04	7.71 ± 0.04	1.68 ± 0.37	76.63 ± 0.53	0.34 ± 0.01
		150	57.82 ± 0.56	5.97 ± 0.01	3.13 ± 0.46	67.06 ± 0.73	0.31 ± 0.01
		180	62.78 ± 0.43	7.83 ± 0.03	2.77 ± 0.35	72.01 ± 0.36	0.28 ± 0.01
MD + GA	12.8	120	60.87 ± 1.03	8.09 ± 0.02	2.81 ± 0.35	58.82 ± 0.56	0.33 ± 0.01
		150	59.07 ± 0.34	7.90 ± 0.02	2.50 ± 0.25	57.53 ± 0.46	0.33 ± 0.01
		180	61.88 ± 0.42	6.78 ± 0.02	2.63 ± 0.32	62.50 ± 0.43	0.31 ± 0.01
	16	120	63.86 ± 2.71	8.83 ± 0.02	1.94 ± 0.27	76.75 ± 0.49	0.35 ± 0.02
		150	65.51 ± 0.26	5.13 ± 0.02	1.79 ± 0.28	56.24 ± 0.32	0.35 ± 0.01
		180	55.95 ± 0.56	8.50 ± 0.03	2.85 ± 0.22	70.44 ± 0.29	0.30 ± 0.01
	19.2	120	43.35 ± 0.58	8.69 ± 0.02	2.05 ± 0.25	72.50 ± 0.85	0.34 ± 0.01
		150	56.86 ± 0.35	6.45 ± 0.02	3.43 ± 0.24	64.32 ± 0.48	0.34 ± 0.01
		180	60.86 ± 0.43	7.23 ± 0.02	2.57 ± 0.18	73.09 ± 0.55	0.29 ± 0.01

BCD = β-cyclodextrin; MD = maltodextrin; GA = gum arabic. Results are expressed as mean ± SD.

Table 2. Influence of carrier type, carrier concentration in feed solution and inlet temperature on the product yield, moisture content, hygroscopicity, solubility and bulk density of produced powders.

	Product Yield (%)	**Moisture (%)**	**Hygroscopicity (g/100 g)**	**Solubility (%)**	**Bulk Density (mL/g)**
Carrier	***p* < 0.01 ***	***p* < 0.01 ***	***p* < 0.01 ***	***p* < 0.01 ***	***p* < 0.01 ***
BCD	36.35 ± 1.56 ^{a}	10.26 ± 0.36 ^{c}	2.26 ± 0.23 ^{b}	37.11 ± 1.22 ^{a}	0.30 ± 0.01 ^{a}
MD	54.92 ± 1.12 ^{b}	4.21 ± 0.13 ^{a}	0.21 ± 0.05 ^{a}	82.89 ± 0.57 ^{c}	0.39 ± 0.03 ^{b}
GA	56.18 ± 2.80 ^{b}	5.34 ± 0.25 ^{ab}	3.06 ± 0.17 ^{b}	70.63 ± 1.63 ^{b}	0.38 ± 0.01 ^{ab}
BCD + GA	56.52 ± 1.26 ^{b}	5.91 ± 0.27 ^{b}	2.86 ± 0.19 ^{b}	68.69 ± 1.47 ^{b}	0.31 ± 0.01 ^{a}
MD + GA	59.62 ± 1.45 ^{b}	7.51 ± 0.23 ^{c}	2.51 ± 0.10 ^{b}	65.80 ± 1.41 ^{b}	0.33 ± 0.00 ^{a}
m(Carrier):V(Extract)	p = 0.32	p = 0.33	p = 0.54	p = 0.38	p = 0.08
12.8	51.42 ± 1.30 ^{a}	6.27 ± 0.33 ^{a}	2.26 ± 0.21 ^{a}	64.69 ± 2.52 ^{a}	0.36 ± 0.02 ^{a}
16	54.85 ± 2.09 ^{a}	6.58 ± 0.41 ^{a}	2.31 ± 0.20 ^{a}	62.16 ± 2.74 ^{a}	0.34 ± 0.01 ^{a}
19.2	51.88 ± 1.98 ^{a}	7.09 ± 0.37 ^{a}	1.97 ± 0.18 ^{a}	68.22 ± 2.10 ^{a}	0.32 ± 0.01 ^{a}
Temperature (°C)	p = 0.77	p < 0.01 *	p < 0.01 *	p = 0.12	p < 0.01 *
120	52.91 ± 2.28 ^{a}	7.67 ± 0.36 ^{b}	1.75 ± 0.14 ^{a}	65.55 ± 2.58 ^{a}	0.35 ± 0.01 ^{b}
150	52.62 ± 1.55 ^{a}	6.35 ± 0.39 ^{a}	1.99 ± 0.20 ^{a}	61.52 ± 2.50 ^{a}	0.36 ± 0.02 ^{b}
180	52.61 ± 1.59 ^{a}	5.92 ± 0.32 ^{a}	2.78 ± 0.22 ^{b}	68.00 ± 2.30 ^{a}	0.31 ± 0.01 ^{a}

Results are expressed as mean ± SE. * Statistically significant variable at $p \leq 0.05$. Values with different letters within columns are statistically different at $p \leq 0.05$. BCD = β-cyclodextrin; MD = maltodextrin; GA = gum arabic; DM = dry matter.

Table 3. Total phenolic content and antioxidant activity of the obtained *Pistacia lentiscus* leaf extract powders.

Carrier	**m(Carrier):V(Extract) (%)**	**Temperature (°C)**	**TPC (mg/g DM)**	**AOA (μmoL Trolox/g DM)**
BCD	12.8	120	129.77 ± 13.17	71.45 ± 8.91
		150	137.44 ± 22.32	168.64 ± 37.56
		180	114.21 ± 20.83	166.00 ± 28.21
	16	120	146.78 ± 21.53	140.54 ± 35.63
		150	125.83 ± 17.92	84.97 ± 9.60
		180	158.04 ± 20.58	89.47 ± 7.52
	19.2	120	147.80 ± 20.43	73.21 ± 27.29
		150	142.17 ± 15.73	97.79 ± 15.57
		180	167.32 ± 15.70	58.54 ± 16.50
MD	12.8	120	101.56 ± 14.82	41.87 ± 5.71
		150	67.93 ± 10.91	22.40 ± 5.05
		180	75.97 ± 13.78	25.07 ± 3.43
	16	120	70.21 ± 15.89	24.04 ± 2.43
		150	65.09 ± 21.74	27.85 ± 7.70
		180	67.82 ± 16.50	26.87 ± 3.04
	19.2	120	71.70 ± 11.67	29.12 ± 4.85
		150	66.93 ± 12.86	31.38 ± 4.22
		180	68.23 ± 9.74	31.94 ± 3.90
GA	12.8	120	107.93 ± 15.54	57.22 ± 12.53
		150	33.92 ± 8.56	25.44 ± 6.85
		180	91.78 ± 12.48	53.93 ± 10.22
	16	120	91.31 ± 11.10	63.65 ± 10.62
		150	111.69 ± 15.56	62.09 ± 13.22
		180	115.23 ± 15.86	61.78 ± 26.06
	19.2	120	174.19 ± 20.87	119.03 ± 11.03
		150	95.92 ± 10.13	41.47 ± 4.37
		180	93.35 ± 17.12	37.24 ± 2.94

Table 3. *Cont.*

Carrier	m(Carrier):V(Extract) (%)	Temperature (°C)	TPC (mg/g DM)	AOA (µmoL Trolox/g DM)
BCD + GA	12.8	120	124.25 ± 16.02	93.33 ± 19.72
		150	111.92 ± 13.82	110.49 ± 9.79
		180	117.27 ± 17.23	120.95 ± 13.56
	16	120	115.73 ± 16.55	46.88 ± 6.15
		150	98.64 ± 16.72	51.30 ± 9.56
		180	175.90 ± 15.83	85.94 ± 5.76
	19.2	120	130.72 ± 15.34	58.89 ± 9.49
		150	143.41 ± 19.37	56.82 ± 10.25
		180	181.44 ± 20.78	108.51 ± 20.76
MD + GA	12.8	120	151.39 ± 11.47	93.02 ± 7.96
		150	138.33 ± 12.21	58.30 ± 10.78
		180	180.83 ± 12.32	71.30 ± 6.86
	16	120	95.81 ± 14.75	40.35 ± 5.58
		150	84.82 ± 14.14	33.63 ± 7.07
		180	169.36 ± 18.11	79.22 ± 2.74
	19.2	120	155.49 ± 15.36	87.00 ± 1.30
		150	154.13 ± 19.56	91.39 ± 6.07
		180	192.88 ± 15.79	85.57 ± 1.22

BCD = β-cyclodextrin; MD = maltodextrin; GA = gum arabic; TPC = total phenolic content; AOA = antioxidant activity. Results are expressed as mean ± SD.

Table 4. Influence of carrier type, carrier concentration and inlet temperature on total phenolic content and antioxidant activity of produced powders.

	TPC (mg/g DM)	AOA (µmoL Trolox/g DM)
Carrier	***p* < 0.01 ***	***p* < 0.01 ***
BCD	141.04 ± 4.29 ^{c}	97.47 ± 8.42 ^{c}
MD	70.25 ± 3.94 ^{a}	26.07 ± 1.51 ^{a}
GA	101.70 ± 7.09 ^{b}	56.76 ± 5.41 ^{b}
BCD + GA	133.25 ± 5.95 ^{c}	78.12 ± 6.13 ^{bc}
MD + GA	140.57 ± 8.31 ^{c}	67.78 ± 4.72 ^{bc}
m(Carrier):V(Extract)	p = 0.03 *	p = 0.39
12.8	108.44 ± 5.60 ^{a}	72.67 ± 6.72 ^{a}
16	112.82 ± 5.68 ^{ab}	59.33 ± 5.02 ^{a}
19.2	130.83 ± 6.99 ^{b}	63.73 ± 4.84 ^{a}
Temperature (°C)	p = 0.06	p = 0.39
120	120.98 ± 4.95 ^{a}	66.64 ± 5.31 ^{a}
150	105.21 ± 5.57 ^{a}	61.22 ± 6.13 ^{a}
180	125.90 ± 7.62 ^{a}	67.87 ± 5.43 ^{a}

Results are expressed as mean ± SE. * Statistically significant variable at $p \leq 0.05$. Values with different letters within columns are statistically different at $p \leq 0.05$. BCD = β-cyclodextrin; MD = maltodextrin; GA = gum arabic; TPC = total phenolic content; AOA = antioxidant activity.

4. Discussion

The product yield of the obtained *Pistacia lentiscus* leaf extract powders ranged from 23.53% (BCD, 19.2%, 180 °C) to 65.51% (MD + GA, 16%, 150 °C) (Table 1), which is similar to the product yield of encapsulated eggplant peel extract (39.58–66.47%) [27] and mulberry leaf extract (38–74%) [28] but lower than the yield of encapsulated nettle leaf extract (64.63–87.23%) [29]. According to Gawalak [30], the product yield obtained at laboratory scale should be at least 50% to achieve satisfactory spray-drying efficiency at industrial scale, implicating that some of the applied process conditions in our study did not meet the required efficiency. As can be seen from the range of results shown in Table 1, the

majority of the powders produced with a yield below 50% were obtained with BCD as a carrier material. Statistical analysis (Table 2) confirmed these observations, showing that the carrier type significantly affected the product yield ($p < 0.01$). The lowest product yield was obtained when BCD was used as a carrier, while no significant difference was observed between the other carriers. Cegledi et al. [29] obtained a higher product yield when BCD or MD was used as a carrier, namely 73.11% and 78.94%, which could be due to the difference in experimental design, regarding the concentration of carriers in the feed solution, which was higher in our study, which probably resulted in lower product yields due to the higher viscosity of the mixture [18].

Moisture content is important for powder stability, flowability, stickiness, microbial growth and the oxidation of bioactive compounds. Powders with moisture content between 4 and 6% are suitable for long-term storage [23], and the moisture content of the obtained powders ranged from 2.89% (MD, 16%, 180 °C) to 12.03% (BCD, 12.8%, 120 °C and BCD, 19.2%, 120 °C) (Table 1). Moisture content was above 6% for powders prepared with BCD and below 6% for those containing MD, and, as shown in Table 2, the difference between the carriers regarding the moisture content was statistically significant ($p < 0.01$). However, compared to our results, the sage extract powders had higher moisture content when GA and MD were used as carriers, and the lowest was seen in BCD powders [31]. In our study, much higher concentrations of BCD were used, and probably many molecules of BCD did not form a complex with a polyphenolic molecule, so more water remained trapped in the molecules of BCD. This observation regarding the higher moisture content within BCD powders can also be correlated to the lower product yield obtained with the same carrier, as elevated moisture content also contributes to the adherence of powder particles on chamber walls, resulting consequently in less product in the collection vessel.

Apart from the carrier type, the inlet temperature also had a significant effect ($p < 0.01$) on the moisture content (Table 2). Increasing the inlet temperature resulted in lower moisture content, which could have been due to the higher temperature gradient between the hot air and the atomized particles, leading to higher heat transfer and thus a higher evaporation rate. Similar results were reported for Moldavian balm extract [32], watermelon juice [25], lemongrass leaf extract [33] and liquorice extract [34].

Hygroscopicity is defined as the ability of powders to bind moisture from the environment and is particularly important for the storage of the product [35]. Hygroscopicity ranged from 0.06 g/100 g (MD, 16%, 120 °C) to 4.40 g/100 g (BCD + GA, 12.8%, 180 °C) (Table 1). The powders can be classified as non-hygroscopic (<10%), slightly hygroscopic (10–15%) and hygroscopic (15–20%) powders [23]. With regard to this classification, the obtained powder samples in our study could be classified as non-hygroscopic. Our results are significantly lower than those of encapsulated aronia berry extract (12.4 to 15.0%) [36] and, to some extent, similar to those of encapsulated green tea leaf extract (3.22 to 5.75%) [20]. Hygroscopicity was significantly affected by the inlet temperature ($p < 0.01$) and the carrier type ($p < 0.01$) (Table 2). Increasing the inlet temperature resulted in higher hygroscopic values, which could be due to the greater water gradient between the powders and the air. Similar results were obtained for powders obtained via the microencapsulation of eggplant peel extract [27] and cupuassu pulp [37]. Moreover, the MD powders had the lowest hygroscopic values, while the GA powders had the highest hygroscopic values. A similar trend was reported by Cegledi et al. [29], where MD and BCD showed higher hygroscopicity than their combinations with GA, which was attributed to the chemical structures of the carriers. Maltodextrin is less hygroscopic and the hygroscopicity is lower at lower DE values, while GA has a more branched structure and free hydroxyl groups that can bind water.

The solubility of powders denotes their potential to form solutions or suspensions in water [15]. In our study, the solubility of *Pistacia lentiscus* leaf extract powders ranged from 27.11% (BCD, 16%, 120 °C) to 86.84% (MD, 19.2%, 180 °C) (Table 1), which is roughly comparable to the solubility of powders containing encapsulated pineapple peel extract with MD, GA or inulin as a carrier (62% to 75%) [38] and the solubility of powders containing

encapsulated *Elsholtzia ciliata* extract (42.5 ± 0.49 to 99.9 ± 0.65%) [21]. As can be seen in Table 2, the carrier type significantly affected the solubility of the powders ($p < 0.01$). The lowest solubility was observed for BCD and the highest for MD powders. The mixture of BCD and MD with GA did not change the solubility statistically significantly. In the spray drying of *Elsholtzia ciliata* extract, BCD powder had the lowest solubility, while the solubility of MD and GA powders was considerably higher [18], which is consistent with our results. The low solubility of BCD powders is due to the limited water solubility of BCD molecules. The molecules bind strongly in the crystal state and intramolecular hydrogen bonds form between the secondary OH groups, making it difficult for the surrounding water molecules to form hydrogen bonds with BCD molecules [39].

Bulk density is an important factor as it determines the size of the storage container and thus the cost of transportation [40]. The bulk density of *Pistacia lentiscus* leaf extract powders ranged from 0.24 g/mL (BCD, 19.2%, 150 °C and BCD, ratio 12.8%, 180 °C) to 0.45 g/mL (MD, 16%, 120 °C and MD, 19.2%, 120 °C) (Table 1), and the parameters that had a statistically significant effect ($p < 0.01$) were the temperature and carrier type (Table 2). The highest inlet temperature resulted in powders with the lowest bulk density, as was also reported for dried eggplant peel extract [27] and liquorice extract [34]. This could be due to the fact that higher temperatures can improve the evaporation rate and thus produce more porous particles [27], resulting in lower bulk density. Regarding the carrier type, GA powders had the highest bulk density, and BCD powders had the lowest bulk density, which is not in agreement with the results obtained for dried sage leaf extract, where the highest bulk density was obtained when BCD was used as a carrier [31]. Due to the lower solubility of BCD, a larger amount of carrier material was used in our study, resulting in higher viscosity of the feeding solution, which probably led to the larger particle size of the powders and thus the lower bulk density [15].

Generally, when observing the overall physicochemical characteristics of the obtained powders, it can be concluded that the most suitable carrier material for the production of encapsulated *Pisaticia lentiscus* powder is MD at the lowest applied concentration and at a temperature of 150 °C. These conditions provided satisfactory powder yields and low moisture content and hygroscopicity of powders with high solubility.

Total phenolics in the obtained powders ranged from 33.92 mg/g DM (GA, 12.8%, 150 °C) to 192.88 mg/g DM (MD + GA, 19.2%, 180 °C) (Table 3). The results were similar to the TPC obtained for mountain tea extract powder (4.9 to 19.4 g/100 g DM) [41] and lower than those obtained for sage extract (13.28 and 29.72 g/100 g DM) [31]. Only the type of carrier had a significant role ($p < 0.01$) and the TPC was highest for BCD, BCD + GA and MD + GA powders, followed by GA powders, and lowest for MD-containing powders (Table 4). The carrier type ($p < 0.01$) and carrier concentration in the feed solution ($p = 0.03$) statistically significantly affected the TPC (Table 4). As can be seen in Table 4, the highest TPC was observed for BCD-containing powders and in descending order in BCD + GA, MD + GA and GA, and it was the lowest for MD-containing powders.

In spray-dried pineapple peel extract, the highest TPC was obtained for MD, followed by GA powders [38], and in spray-dried mountain tea extract, the highest TPC was obtained for BCD powders, followed by GA powders, but the difference was not statistically significant [41]. The difference between the results could be due to the phenolic composition and content of the microencapsulated extracts. For example, the carrier mixture of GA and MD was reported to improve the retention of hydrophobic molecules, which was attributed to the hydrophobic protein fraction of GA that gives GA an emulsifying ability to retain hydrophobic molecules [42]. When the extract contains hydrophobic molecules, GA encapsulates more molecules than MD. The high result obtained for BCD powder could be due to the formation of inclusion complexes between BCD molecules and phenolic compounds, which increase their stability to heat [17].

Antioxidant activity ranged from 22.40 (MD, 12.8%, 150 °C) to 168.64 (BCD, 12.8%, 150 °C) μmoL Trolox equivalent/g DM (Table 3). The carrier type statistically significantly ($p < 0.01$) affected the AOA (Table 4). The powders with the highest AOA were the powders

with the highest phenolic content (BCD, BCD + GA and MD + GA) and, accordingly, the lowest AOA was measured for MD powders. These results are in agreement with those obtained for encapsulated eggplant peel extract [27], where the powders with the highest antioxidant capacity had the highest TPC, although this was achieved with MD as a carrier.

Both TPC content and AOA were higher in powders produced from mixtures of different carrier materials, namely BCD + GA and MD + GA, and from pure BCD. The reason for the better phenolic-related properties of powders containing BCD and mixtures with GA may lie in the composition of *Pistacia lentiscus* polyphenols, containing a high proportion of flavonoids [3], which, due to their slightly hydrophobic planary structure, which enables their interactions with lipid membrane layers, tend to be retained better in hydrophobic BCD- and GA-containing capsules [43]. In order to obtain a general conclusion on the spray-drying conditions that enable both high TPC and AOA retention, a higher carrier concentration should be applied due to the higher TPC amount obtained at 19.2%, while the spray-drying process could be performed at the lowest evaluated temperature as it did not affect the final amount of phenolics or their AOA.

When compared with the non-encapsulated extract (TPC = 279.53 ± 21.7 mg/g DM, AOA = 374.6 ± 31.4 μmoL Trolox equivalent/g DM), the application of 19.2% of BCD as a carrier at a temperature 120 °C enabled TPC retention of approximately 53%, with the retention of approximately 20% AOA. The TPC retention is lower than that obtained by Gaćina at al. for the microencapsulation of blackthorn flower water extract (87.87%) [44] and in the lower range of the TPC retention results of microencapsulated *C. paliurus* extracts (56.23% to 90.31%) [10]. Chen et al. (2021) explained the difference in TPC retention as a result of the different extraction solvents applied for the preparation of liquid extracts prior to spray drying. Accordingly, ethanol extracts are more heat-sensitive during spray drying, while water extracts contain polysaccharides, which may provide some additional protection to phenolic compounds during the drying process [10]. Hence, the lower retention can be related to the ethanolic *Pistacia lentiscus* extract used for encapsulation.

5. Conclusions

In this study, the spray-drying encapsulation of *Pistacia lentiscus* leaf extract was evaluated in terms of the carrier type, its concentration and the drying temperature in order to obtain a stable, high-quality product with high retention of phenolics and high antioxidant activity. The type of carrier material used for encapsulation was found to be the most crucial parameter for the final product design as it affected both the physical characteristics of powders and the TPC and AOA.

The powders obtained using MD and GA showed better physical properties compared to the BCD powders, which had the lowest product yield, solubility and bulk density and higher moisture content. On the other hand, BCD and carrier mixtures containing GA ensured better retention of phenolics and antioxidant activity for the encapsulated *Pistacia lentiscus* leaf extract. Additionally, for the better preservation of bioactive compounds, a higher proportion of carrier material in the feed solution was necessary, while a temperature effect was not expressed. Thereby, for the final product design, this study recommends the application of carrier mixtures containing GA as the most suitable carrier material that can provide both satisfactory physicochemical characteristics and phenolic retention in encapsulated *Pistacia lentiscus* extract, at a concentration of 19.2% and temperature of 150 °C. These results and observations provide a basis for the development of encapsulated *Pistacia lentiscus* leaf extract that retains a high amount of valuable polyphenols in a stable, powdered form and thereby represents a worthy ingredient for value-added or functional food products and nutraceuticals. Future studies should, however, be directed towards the evaluation of the stability and degradation kinetics of encapsulated *Pistacia lentiscus* phenolic compounds during processing and storage.

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