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Optimization of Microwave-Assisted Extraction from Peppermint (*Mentha piperita* L.) Using Ethanol and β-Cyclodextrin as Green Solvents

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Abstract

This study explores the use of microwave-assisted extraction of bioactive compounds from peppermint (*Mentha piperita* L.) using aqueous ethanolic and β-cyclodextrin solvents. The experiments were performed according to Response Surface Methodology, and the central composite design was employed, with the independent variables being treatment time, microwave power and solvent concentration. During the research, the total polyphenol content (TPC, expressed as gallic acid equivalents, GAE), total flavonoid content (TFC, expressed as quercetin equivalents, QUE), antioxidant capacity (AC) using the FRAP (Ferric Reducing Antioxidant Power, expressed as ascorbic acid equivalents, ASE) and DPPH (2,2-diphenyl-1-picrylhydrazyl) methods, and total menthol content (TMC, expressed as menthol equivalents, MEE) were examined. For the ethanolic extraction, the optimum conditions were 571 W, 7.3 min, and 10% v/v. The following results were obtained for these parameters: TPC, 1025 µg GAE/mL; TFC, 66.69 µg QUE/mL; FRAP, 3786.24 µg ASE/mL; DPPH, 80.24%; and TMC, 62.63 µg MEE/mL. In the case of β-cyclodextrin, the optimum conditions are 800 W, 11.81 min, and 1.70 mM. In this case, the results are as follows: TPC, 949.29 µg GAE/mL; TFC, 90.75 µg QUE/mL; FRAP, 4275.54 µg ASE/mL; DPPH, 89.81%; and TMC, 84.99 µg MEE/mL. β-cyclodextrin increased the flavonoid, antioxidant and menthol content, although there were no significant differences between the concentrations. The use of ethanol had a greater effect on the polyphenol content. β-cyclodextrin proved to be an effective green alternative solvent for peppermint.

Keywords: peppermint; microwave-assisted extraction; ethanol; β-cyclodextrin; antioxidant capacity; polyphenol content; flavonoid content; menthol content

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1. Introduction

Peppermint (*Mentha piperita* L.) is one of the best-known, most popular and widely used medicinal plants, thanks to its characteristic flavour, essential oil content, high menthol content, and significant antimicrobial and antioxidant activity [1,2]. Not only is peppermint consumed as tea, but it is also used in many industries, such as the food, pharmaceutical, cosmetic, and chemical industries [3,4]. Peppermint is native to Europe and the Middle East, but today it is widely cultivated in the United States, China, India, the countries of the former Soviet Union, as well as Hungary, France, Iran, and Italy [4]. According to data from the United States Department of Agriculture National Agricultural Statistics Service [5],

annual peppermint production in the United States was 2.39 million pounds in 2024, which illustrates its economic and industrial importance.

The industrial processing of herbs includes the production of filter teas, which generates a significant amount of by-products and waste. These by-products can account for 10–40% of the initial quantity. One of the most important by-products is fine-grained "herbal powder," which also contains valuable bioactive compounds [6]. Therefore, it is worthwhile to explore alternative methods of processing these "herbal powders," including mint, and to optimize the extraction process required for this.

Peppermint is rich in many minerals, such as Na, Mg, K, Ca, Cr, Fe, Co, Cu, Zn, and Se [7]. In addition, it contains between 0.5% and 4% essential oil [2]. Using gas chromatography–mass spectrometry analysis, ten main compounds of peppermint essential oil have been identified, including menthol (30.0–55.0%), menthone (14.0–32.0%), menthyl acetate (2.8–10.0%), isomenthone (1.5–10.0%), menthofuran (1.0–9.0%), limonene (1.0–5.0%), 1,8-cineole (3.5–4.0%), pulegone (max. 4.0%), isopulegol (max. 0.2%), and carvone (max. 1.0%) [8,9].

Peppermint also has a relatively high content of polyphenols and antioxidants. The polyphenols found in peppermint are mainly in the form of flavonoids and phenolic acids [10]. However, since these compounds are likely to break down at high temperatures, consuming peppermint in the form of tea limits its beneficial effects. For this reason, traditional heat extraction is not the best choice for the effective extraction of the active ingredients of peppermint. Recently, modern alternative methods, such as microwave-assisted extraction (MAE) [11–13], have become increasingly popular alongside traditional extraction methods. This method is widely used in the food, pharmaceutical, and cosmetic industries. The MAE method is based on the fact that under the influence of an electric field, the partial negative and positive charges of polar molecules rotate in the direction of the field [14], and the resulting friction converts electromagnetic energy into heat [15]. This provides rapid energy transfer to the entire volume of the solvent and solid plant matrix, which are then heated efficiently and homogeneously. As the water in the plant matrix absorbs the microwave energy, this leads to heating of the plant tissues and breakdown of the cell wall structure, thereby promoting the desorption of chemicals from the matrix [15,16]. MAE can reduce process times and solvent use [17], or even eliminate solvent use altogether [18], while increasing extraction yields [19]. More studies have shown that MAE is effective in extracting a number of bioactive compounds (for example flavonols and phenolic acids) of natural compounds from plant by-products [17,20–23].

Based on the principles of Green Chemistry, there is a great need to develop environmentally friendly extraction processes using bio-based alternative solvents instead of traditional petroleum-based volatile solvents (e.g., hexane, isopropyl alcohol, petroleum ether, etc.), which pose a serious environmental risk and are not suitable for use in the food industry due to their toxic nature [13,21,24,25]. In optimizing the extraction process, solvents can not only increase efficiency but are also important in terms of sustainability and health protection, especially when the goal is food industrial application. In order for a solvent to be designated as 'green', it must be derived from renewable feedstock and must not persist for an extended period within the environment, and it must not result in the generation of hazardous by-products during its preparation or processing [25]. Green solvents include pure water, ethanol and β-cyclodextrin.

The impact of pure water is limited to moderately polar compounds, which reduces extraction efficiency [26]. Pure ethanol is suitable for extracting polar and moderately polar compounds such as polyphenols, flavonoids, alkaloids, and saponins [20,27]. The combination of two solvents (binary solvent) allows for the extraction of a wider spectrum of polyphenols, resulting in higher yields and bioactivity than when using water or

ethanol alone [28,29]. β-cyclodextrin is an increasingly researched solvent. It can be used to efficiently extract certain bioactive substances, such as flavonoids and some essential oil components [21,26]. Its mechanism is based on the formation of inclusion complexes, through which "guest molecules" are incorporated into the weakly apolar cavity of β-cyclodextrin, thereby improving the solubility and stability of these compounds [30,31]. This solvent can be considered an alternative to traditional solvent extraction. In previous research, β-cyclodextrin has been used as a solvent in conventional extraction processes from mint [21], whereas its combination with MAE has not yet been researched for peppermint. Combining the cell wall disruption and enhanced mass transfer effects of MAE with the inclusion of the complex-forming ability of β-cyclodextrin may provide an effective approach for more efficient extraction and stabilization of sensitive compounds, such as essential oil components (e.g., menthol). Previous studies have mainly focused on the combination of β-cyclodextrin and ultrasound-assisted extraction (beetroot [32], chokeberry [33], grape pomace [34], apple pomace [35], tea leaves [36], and taro leaves [37]), while recent research has begun to investigate its application with MAE in various plant matrices (e.g., turmeric [38], green rooibos [26], brown macroalgae [39], and harpagophytum [40]). However, to our knowledge, the combined effect of MAE and β-cyclodextrin as a solvent has not yet been investigated in the case of peppermint.

The aim of the current research is to determine the optimal extraction method for peppermint microencapsulation and to test solvents that not only play a role in the extraction of active ingredients but also in the production of instant tea powders.

2. Materials and Methods

2.1. Materials

The dried chopped peppermint (*Mentha piperita folium*) used in this study was procured from Gyógyfű Gyógynövény Forgalmazó Kft (Sóskút, Hungary). The mint extracts were produced using a solid–liquid extraction procedure. A ratio of 10 g of peppermint/300 mL of solvent was used during the extractions. The solvents applied in this study included an aqueous ethanol solution (C_2H_6O) at concentrations of 10, 35, and 60% v/v, as well as an aqueous β-cyclodextrin solution at concentrations of 1 mM, 2 mM, and 3 mM.

2.2. Chemicals

Sodium acetate trihydrate, ethanol (96%), and Ethanol absolute (>99.7%) were purchased from VWR Chemicals (Budapest, Hungary). ABTS, DPPH, Gallic acid, Folin & Ciocalteu's phenol reagent, L-ascorbic acid, Potassium persulfate, Quercetin hydrate and 2,4,6-tris(2-pyridyl)-s-triazine (TPTZ) were purchased from Sigma–Aldrich (Gillingham, UK). Aluminium chloride (99%) was purchased from Acros Organics (Fair Lawn, NJ, USA). Acetic acid (99.8%) and Sulfuric acid (96%) were purchased from Lach-Ner (Neratovice, Czech Republic), and Hydrochloric acid (37%) was purchased from Carlo Erba Reagents S.A.S. (Val de Reuil, France). Iron(III) chloride hexahydrate was purchased from Reanal (Budapest, Hungary). β-cyclodextrin (98%), L-menthol 99.5%, Vanillin (99%) were purchased from Thermo Scientific (Waltham, MA, USA). The chemicals utilized in this study were of analytical reagent grade.

2.3. Methods

2.3.1. Extraction Method

Microwave-assisted solid–liquid extraction was used during our research. This was carried out using a Specs Electrolux EMM 2005 microwave oven. In order to prevent the solvent from overheating and evaporating, pulse mode was used for the process [15,17,41,42], during which the power was pulsed, as determined by preliminary experiments. At 100 W

of power, an 8 min activation and 2 min inactivation cycle (using an ice-water bath) was alternated during the measurement. At 450 W, a 1 min activation and 2 min inactivation cycle was alternated. At 800 W of power, there was a 0.5 min activation and 2 min inactivation cycle. During microwave-assisted extraction, the temperature of the mint extraction was monitored and, due to the applied pulsed operation with intermittent cooling cycles, did not exceed 42 °C under any experimental condition.

Filtering using a vacuum filter (MZ 2C vacuum pump, Vacuubrand GmbH & Co., Ltd., Wertheim, Germany) with general-purpose filter papers (MN 604 D = 90 mm, Macherey-Nagel GmbH & Co. KG, Düren, Germany) was performed to remove any suspended matter. It was then centrifuged at 6000 rpm for 20 min using a Hermle Z 206 A compact centrifuge (HERMLE Labortechnik GmbH, Wehingen, Germany) to separate any remaining suspended matter. The sample was stored in Eppendorf tubes in a freezer until analytical testing (Figure 1).

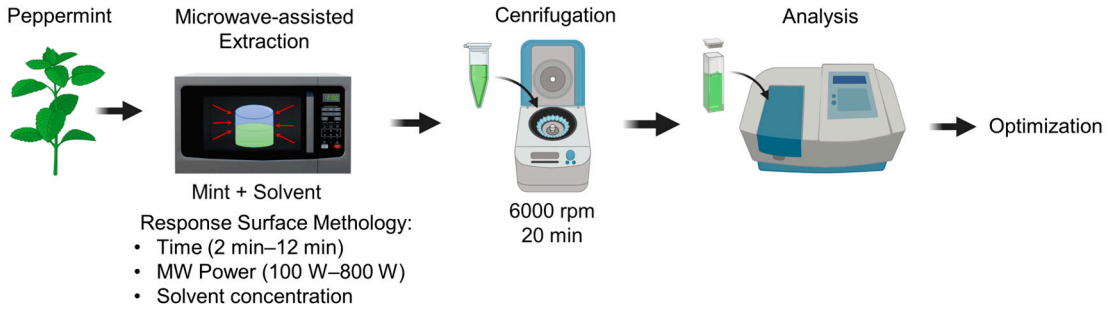


Figure 1. The sequence of experimental procedures used in this research (illustration created with BioRender.com (https://www.biorender.com/)).

2.3.2. Determination of Antioxidant Capacity (AC)

FRAP Assay

Antioxidant capacity was determined using a modified method developed by Benzie and Strain [43]. The method exploits the iron-reducing capacity of the FRAP reagent, in which 2,4,6-tri(2-pyridyl)-s-triazine (TPTZ) acts as a chelating compound in the reaction. Ascorbic acid was used as the standard reference. Briefly, the FRAP reagent was prepared from 250 mL of 0.3 M of acetate buffer, 25 mL of 10 mM of the TPTZ solution, and 25 mL of 20 mM of iron(III) chloride. A total of 40 µL (3 replicates) of extract was mixed with 10 µL of distilled water and 1500 µL of the FRAP reagent. Exactly 5 min after mixing, the absorbance of the sample was measured at 593 nm utilizing a Jasco 7850 UV-Vis spectrophotometer (Jasco International Co., Ltd., Tokyo, Japan) for absorbance determination. A calibration curve was prepared using ascorbic acid in distilled water. Antioxidant capacity is expressed in ascorbic acid equivalents (µg ASE/mL). AC was calculated using the following formula:

$$AC(FRAP) = \frac{A}{tg\alpha_1} \cdot \frac{S\ total}{S\ sample} \cdot DF \quad (1)$$

where A is the measured absorbance; $tg\alpha_1$ is the slope of the calibration curve; $S\ total$ is the total volume of the sample solution (µL); $S\ sample$ is the actual volume of the sample (µL); and DF is the dilution factor.

DPPH Assay

Antioxidant capacity was determined using the α,α-diphenyl-β-picrylhydrazyl (DPPH) method described by Brand-Williams et al. [44]. The method is based on the reaction of antioxidant-containing compounds with radicals. Briefly, 3.9 mL of a methanol DPPH solution (0.0236 g of DPPH powder dissolved in 1000 cm^3 of methanol) was added

to 0.1 mL portions (3 replicates) of the mint extracts. The mint samples were necessarily diluted to be within the calibration range. After thorough homogenization, the mixture was incubated for 20 min in the dark at room temperature. The absorbance was measured at 517 nm using a UV-Vis spectrophotometer (Jasco 7850, Tokyo, Japan). Antioxidant capacity is expressed in radical scavenging activity and is given as the percentage inhibition, which indicates the percentage of the radical solution of a given concentration that the tested sample is able to neutralize. This is calculated as follows:

$$AC(DPPH) = \frac{(A_0 - A)}{A_0} \cdot 100 \quad (2)$$

where A_0 is the absorbance of the radical solution (control), and A is the absorbance of the reaction mixture (sample with DPPH).

2.3.3. Determination of Total Polyphenol Content (TPC)

The total phenolic content was determined using the method described by Singleton and Rossi [45], which involved the use of the Folin–Ciocalteu reagent. The method is based on the reducing capacity of phenolic compounds, which causes a colour change in the reagent. Gallic acid was used as the standard reference. Briefly, 10 µL (3 replicates) of extract was mixed with 240 µL of diluent (a mixture of 80:20 methyl alcohol and distilled water) and 1250 µL of the Folin–Ciocalteu solution (a 1:10 mixture of reagent and diluent). The mixture was allowed to stand for 1 min, and then 0.7 M of an aqueous Na_2CO_3 solution was added. This was followed by incubation at 50 °C for 5 min, after which the absorbance was measured at 760 nm. A calibration curve was prepared using gallic acid in methanol. All phenol values are expressed in gallic acid equivalents (µg GAE/mL). TPC was calculated using the following formula:

$$TPC = \frac{A}{tg\alpha_2} \cdot \frac{S\ total}{S\ sample} \cdot DF \quad (3)$$

where A is the measured absorbance; $tg\alpha_2$ is the slope of the calibration curve; *S total* is the total volume of the sample solution (µL); *S sample* is the actual volume of the sample (µL); and *DF* is the dilution factor.

2.3.4. Determination of Total Flavonoid Content (TFC)

The total flavonoid content was determined using a modified method developed by Akbay et al. [46] following the description by Tistantini and Amalia [47]. The determination is based on the complexing reaction between flavonoids and aluminium chloride ($AlCl_3$). Quercetin was used as the standard reference. Briefly, 25 µL (3 replicates) of the samples were mixed with 2375 µL of distilled water, 50 µL of 1 M of a potassium acetate solution, and 50 µL of a 10% w/v aluminium chloride solution. The mixture was then homogenized using a vortex mixer for 60 s. The homogenized mixture was incubated at room temperature in the dark for 30 min. The absorbance of the reaction mixture was measured using a UV-Vis spectrophotometer (Jasco 7850, Tokyo, Japan) at 415 nm. A calibration curve was prepared using a quercetin solution (0.001 g/cm^3 of an ethanol–quercetin mixture). The total flavonoids are expressed as quercetin equivalents (µg QUE/mL), and the TFC was calculated accordingly. TFC was calculated using the following formula:

$$TFC = \frac{A}{tg\alpha_3} \cdot \frac{S\ total}{S\ sample} \cdot DF \quad (4)$$

where A is the measured absorbance; $tg\alpha_3$ is the slope of the calibration curve; *S total* is the total volume of the sample solution (µL); *S sample* is the actual volume of the sample (µL); and *DF* is the dilution factor.

2.3.5. Determination of Total Menthol Content (TMC)

The total menthol content (TMC) was determined using a modified method developed by Dangwal [48] and Kushwah et al. [49]. This is a colorimetric method based on the colour change that occurs when the reaction product of menthol and vanillin is diluted in a concentrated sulfuric acid medium. Menthol was used as the standard reference. Briefly, 100 µL (3 replicates) of the tested extracts were added to 250 µL of a 1% w/v vanillin reagent (1 g of vanillin dissolved in 100 cm^3 of concentrated H_2SO_4). The mixture was then incubated at room temperature for 5 min. Subsequently, 2150 µL of distilled water was added to the mixture, followed by incubation for 5 min. The absorbance was measured at 525 nm using a UV-Vis spectrophotometer (Jasco 7850, Tokyo, Japan). The total menthol is expressed as menthol equivalents (µg MEE/mL). TMC was calculated using the following formula:

$$TMC = \frac{A}{tg\alpha_4} \cdot \frac{S\ total}{S\ sample} \cdot DF \quad (5)$$

where A is the measured absorbance; $tg\alpha_4$ is the slope of the calibration curve; *S total* is the total volume of the sample solution (µL); *S sample* is the actual volume of the sample (µL); and *DF* is the dilution factor.

2.3.6. Statistical Analysis

Three parallel measurements were performed per sample, and the results are expressed as the mean ± standard deviation using the Microsoft® Excel software. Response Surface Methodology (RSM) was used to evaluate the experimental data, using a Face-Centred Central Composite Design (α = 1). This produces a design where axial points coincide with factorial points, meaning that each factor has only three levels. Table 1 provides a detailed description of the independent variables and their respective levels. The variables under consideration are as follows: treatment time, microwave power and solvent concentration. A randomized CCD with 20 experimental runs was employed. Centre points were repeated six times to get a good estimate of experimental pure error. A quadratic polynomial model was fitted to all response variables. Prior to modelling, the FRAP response was subjected to a square-root transformation to improve normality and stabilize variance, as indicated by model diagnostics. Modelling and optimization calculations were performed using the demo version of the Stat-Ease® 360 Design Expert software. The statistical significance level was set at p = 0.05.

Table 1. Range of coded and actual values of extraction parameters for central composite design (α = 1) when using ethanol and β-cyclodextrin as a solvent.

	Treatment Time (min)	Microwave Power (W)	Solvent Concentration	
			Ethanol (% *v/v*)	β-Cyclodextrin (mM)
Min (−1)	2	100	10	1
Centre (0)	7	450	35	2
Max (+1)	12	800	60	3

3. Results and Discussion

3.1. RSM of Ethanol Extracts

The antioxidant activity (FRAP and DPPH), total polyphenol content (TPC), total flavonoid content (TFC) and total menthol content (TMC) obtained from peppermint extracts using an ethanol solvent prepared by MAE are represented by response surface models derived from a central composite design, which are the following:

$$\text{Sqrt}(\text{AC}(\text{FRAP})) = 56.227 - 0.271 \cdot A + 0.031 \cdot B - 0.271 \cdot C + 0.018 \cdot AC - 0.000028 \cdot B^2 \quad (6)$$

$$\text{TPC} = 985.275 + 14.429 \cdot A - 0.142 \cdot B - 0.363 \cdot C + 0.022 \cdot AB + 0.342 \cdot AC + 0.003 \cdot BC - 1.967 \cdot A^2 - 0.118 \cdot C^2 \quad (7)$$

$$\text{TFC} = 51.953 + 0.599 \cdot A + 0.029 \cdot B + 0.074 \cdot C + 0.017 \cdot AC - 0.000023 \cdot B^2 - 0.006 \cdot C^2 \quad (8)$$

$$AC(DPPH) = 60.308 - 0.252 \cdot A + 0.010 \cdot B - 0.535 \cdot C + 0.033 \cdot AC - 0.000087 \cdot C^2 \quad (9)$$

$$TMC = 88.113 - 1.313 \cdot A - 0.010 \cdot B - 1.920 \cdot C + 0.059 \cdot AC + 0.00053 \cdot BC + 0.016 \cdot C^2 \quad (10)$$

where the A is the treatment time (2 min–12 min), B is the microwave power (100 W–800 W), and C is the solvent concentration (10% v/v 60% v/v) in the evaluated ranges.

As illustrated in Table 2, the analysis of variance (ANOVA) for the response variables is based on the experiments that were performed. The models obtained were statistically significant ($p < 0.0001$), and the tests for lack of fit were not significant, thus confirming the adequacy of the models. During the evaluation of the model performance, the correlation coefficients were found to be R^2 = 0.96, 0.84, 0.95, 0.94, and 0.87 for AC(FRAP), TPC, TFC, AC(DPPH), and TMC, respectively, which indicates good model adequacy and predictive capability. Pearson's correlation coefficients were used to analyse the relationship between the bioactive substances under investigation, which exhibited a strong correlation, except for TMC. (Table 3).

Table 2. For the ethanol-based peppermint extracts, results of ANOVA for reduced response surface quadratic model.

Model	Sum of Squares	df	Mean Square	F-Value	*p*-Value	Residual (SS)	Lack of Fit (SS)
AC(FRAP) (µg ASE/mL)	305.54	5	61.11	14.68	<0.0001	58.28	36.75
TPC (µg GAE/mL)	275,126.43	8	34,390.80	29.42	<0.0001	12,856.56	5607.92
TFC (µg QUE/mL)	1090.76	6	181.79	40.45	<0.0001	58.43	42.87
AC(DPPH) (%)	1472.08	5	294.42	40.90	<0.0001	86.38	39.13
TMC (µg MEE/mL)	1452.82	6	242.14	14.155	<0.0001	205.31	176.42

The measured responses (TPC, TFC, AC(DPPH) and AC(FRAP)) were significantly affected by extraction time, microwave power and solvent concentration. Among the interaction members, the time–solvent concentration interaction (AC) had a significant and positive effect on all responses. In the TPC test, the other interactions (AB and BC) had an even lower effect. Several quadratic terms, such as time^2 (A^2), power^2 (B^2), and concentration^2 (C^2), were also important, indicating the non-linear nature of the extraction process.

Table 3. For the ethanol-based peppermint extracts, Pearson's correlation coefficients between AC(FRAP), TPC, TFC, AC(DPPH) and TMC.

	AC(FRAP) (μg ASE/mL)	**TPC (μg GAE/mL)**	**TFC (μg QUE/mL)**	**AC(DPPH) (%)**	**TMC (μg MEE/mL)**
AC(FRAP) (μg ASE/mL)	1	0.861	0.861	0.831	0.221
TPC (μg GAE/mL)		1	0.891	0.850	0.112
TFC (μg QUE/mL)			1	0.891	0.207
AC(DPPH) (%)				1	0.182
TMC (μg MEE/mL)					1

Figure 2 shows the graphical representation of the models. The response surface plots demonstrate the combined impact of microwave power and extraction time on the parameters investigated for the peppermint extracts obtained using an ethanol solvent. The third parameter that was varied in the experimental design, which represents the solvent concentration, was set to 35% v/v in all 3D plots. The diagrams clearly show that the material under investigation does not change linearly within the range of dependent variables examined. In all cases, curvature is observed, which indicates cross effects in addition to the main effects. Increasing the microwave power and time can increase response values up to a certain point. This is caused by the destruction of the cell wall structure due to microwaves [15,16] and the prolonged contact between the plant matrix and the extract [20]. After that a slight decline or plateau is apparent. These data also suggest that phenolic compounds may degrade under non-ideal processing conditions due to excessive temperature, thereby reducing extraction efficiency [27,50,51].

The AC of the peppermint samples tested ranged from 1865.87 ± 104.65 to 3829.88 ± 32.87 μg ASE/mL, as determined by the FRAP method. Similarly, the range of the DPPH method was from 33.83 ± 0.54 to 76.16 ± 1.78%. The tested TPC ranged from 613.7 ± 30.75 to 1037.53 ± 24.64 μg GAE/mL, the TFC from 37.98 ± 3.23 to 71.46 ± 1.48 μg QUE/mL and the TMC from 34.45 ± 0.42 to 75.21 ± 6.06 μg MEE/mL.

The maximum yields of the examined bioactive compounds, along with the associated parameters, were determined separately and are presented in Table 4. This indicates that the independent variables were maintained within the experimental range under investigation, while the response variable was set to "maximization" and the remaining responses were excluded from the desirability function during the optimization process. This methodological approach enabled us to independently ascertain the theoretical maximum of each response.

Each column corresponds to the independent maximization of one response variable. The optimal MAE parameters for each response are delineated in the initial three rows of this table: time, microwave power, and solvent concentration. The subsequent rows of the table present the maximum anticipated values of bioactive compounds under these optimal conditions. The table illustrates the compounds with the highest predicted response values.

The maximum collective yield of the bioactive compounds under investigation (TPC, AC(FRAP), AC(DPPH), TMC and TFC) was achieved simultaneously with a treatment time of 7.30 min, a microwave power of 571.03 W, and a solvent concentration of 10.00% v/v. The predicted values in this instance were as follows: The TPC was found to be 1025.00 μg GAE/mL, the TFC 66.69 μg QUE/mL, the AC(FRAP) 3786.24 μg ASE/mL, the AC(DPPH) 80.24% and the TMC 62.63 μg MEE/mL.

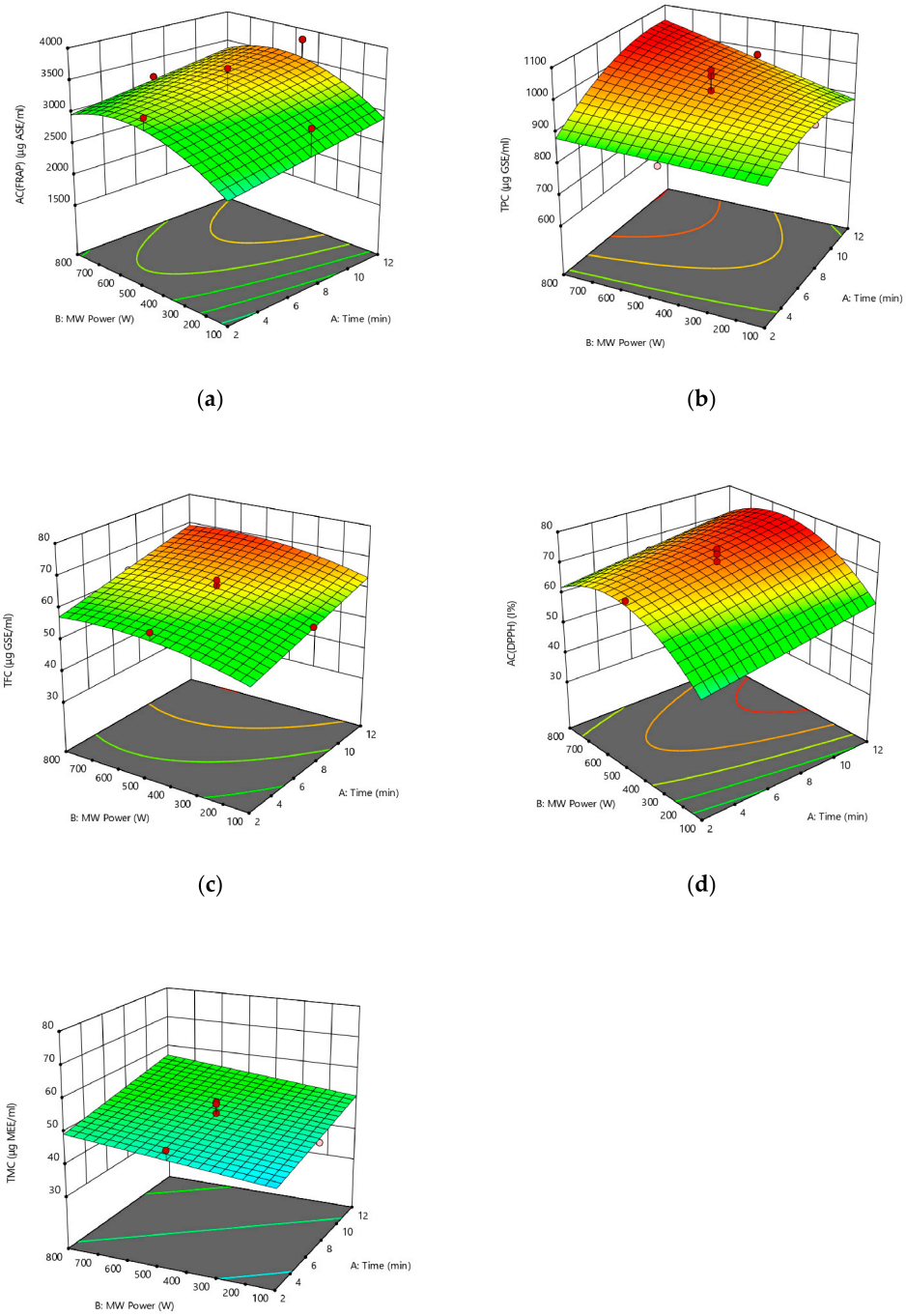


Figure 2. For the ethanol-based peppermint extracts, 3D diagrams showing the effect of the independent (process) variables on the responses considered, with the solvent concentration set at medium (35% v/v): (**a**) the effect on AC(FRAP), (**b**) the effect on TPC, (**c**) the effect on TFC, (**d**) the effect on AC(DPPH) and (**e**) the effect on TMC.

The optimal microwave power identified in the present study is consistent with Pavlić et al. [52], who, using an aqueous ethanol system, observed that a microwave power of around 600 W resulted in the highest TPC and TFC recovery during peppermint MAE. Additionally, the authors noted that the highest amount of phenolic compounds were released from the plant material during the first 5 min of the extraction process, and after 10 min, the process shifted to a slower diffusion-controlled stage, where the extraction

yields no longer increased noticeably. The optimized extraction time obtained in the present study falls precisely within this time range.

Table 4. For the ethanol-based peppermint extracts, the maximum yields for each were response-considered, and the optimal conditions were predicted by desirability.

		Optimized Response				
		AC(FRAP)	**TPC**	**TFC**	**AC(DPPH)**	**TMC**
Optimal Conditions	Time (min)	2.51	9.79	12.00	10.96	11.95
	MW Power (W)	594.52	776.14	631.37	552.55	768.09
	Solvent concentration (% v/v)	10.16	19.87	21.46	11.09	59.52
Maximum Predicted Value	AC(FRAP) (μg ASE/mL)	3830.70	3479.10	3641.07	3736.85	3267.37
	TPC (μg GAE/mL)	969.82	1057.40	1022.69	1005.77	920.27
	TFC (μg QUE/mL)	63.06	68.10	71.23	69.58	61.32
	AC(DPPH) (%)	79.62	73.14	78.26	80.40	68.83
	TMC (μg MEE/mL)	65.78	55.65	54.90	59.41	75.24

Similarly, Dai et al. [19] determined the optimal microwave power to be around 500 W for menthol extraction, using ethanol, hexane, and their mixtures as solvents. This value is very close to the optimal power determined in the present study. However, in our experiments, the optimal extraction yield was achieved in a much shorter treatment time, indicating that the parameters used ensured a faster and more efficient extraction process. The reduced extraction time also makes MAE more energy-efficient and may reduce the processing time and energy input. The simultaneous optimization of phenolic content, antioxidant activity and menthol recovery under low ethanol concentration provides a balanced extraction strategy within the investigated MAE system and allows for a more comprehensive evaluation of peppermint extractability than approaches focusing on individual response variables.

3.2. RSM of β-Cyclodextrin Extracts

Similarly, RSM was used to analyse the impact of extraction parameters on peppermint extracts produced using a β-cyclodextrin solution. These are the regression equations for the model:

$$\mathrm{Sqrt(AC(FRAP))} = 46.685 - 0.334 \cdot A + 0.039 \cdot B + 2.597 \cdot D - 0.0032 \cdot BC + 0.079 \cdot A^2 - 0.000031 \cdot D^2 \quad (11)$$

$$\mathrm{TPC} = 774.430 + 9.275 \cdot A + 0.0804 \cdot B + 0.573 \cdot D \quad (12)$$

$$\mathrm{TFC} = 44.649 + 5.087 \cdot A + 0.0064 \cdot B + 16.286 \cdot D - 0.249 \cdot A^2 - 4.171 \cdot D^2 \quad (13)$$

$$AC(DPPH) = 60.906 + 6.293 \cdot A + 0.016 \cdot B - 1.673 \cdot D - 0.396 \cdot A^2 \quad (14)$$

$$TMC = 63.197 + 1.191 \cdot A + 0.023 \cdot B - 6.385 \cdot D \quad (15)$$

where the A is the treatment time (2 min–12 min), B is the microwave power (100 W–800 W), and D is the solvent concentration (1 mM–3 mM) in the evaluated ranges.

As shown in Table 5, ANOVA was performed on the response variables based on the experiments conducted. All models were statistically significant ($p < 0.0001$, except for TMC, $p = 0.0002$), while lack-of-fit tests were not, confirming the models' reliability. Evaluation of model performance revealed correlation coefficients of $R^2 = 0.85$, 0.93, 0.87, 0.82 and 0.85 for AC(FRAP), TPC, TFC, TMC and AC(DPPH), respectively, indicating the model's strong predictive capability. Pearson's correlation test also supports this (Table 6).

Table 5. For the β-cyclodextrin-based peppermint extracts, results of ANOVA for reduced response surface quadratic model.

Model	Sum of Squares	df	Mean Square	F-Value	*p*-Value	Residual (SS)	Lack of Fit (SS)
AC(FRAP) (µg ASE/mL)	197.51	6	32.92	21.09	<0.0001	15.61	7.24
TPC (µg GAE/mL)	28,233.24	3	9411.08	17.95	<0.0001	6814.56	4433.71
TFC (µg QUE/mL)	11,189.24	5	223.749	17.56	<0.0001	165.64	54.81
AC(DPPH) (%)	916.60	4	229.15	17.14	<0.0001	160.42	52.25
TMC (µg MEE/mL)	438.58	3	146.19	17.09	0.0002	94.10	61.10

Table 6. For the β-cyclodextrin-based peppermint extracts, Pearson's correlation coefficients between AC(FRAP), TPC, TFC, AC(DPPH) and TMC.

	AC(FRAP) (µg ASE/mL)	TPC (µg GAE/mL)	TFC (µg QUE/mL)	AC(DPPH) (%)	TMC (µg MEE/mL)
AC(FRAP) (µg ASE/mL)	1	0.726	0.691	0.426	0.660
TPC (µg GAE/mL)		1	0.897	0.853	0.635
TFC (µg QUE/mL)			1	0.826	0.710
AC(DPPH) (%)				1	0.547
TMC (µg MEE/mL)					1

In this case, the measured responses were also significantly influenced by the extraction time and microwave power, but the solvent concentration did not show a significant effect on the TPC, TFC, TMC and DPPH measurements when β-cyclodextrin was used. In contrast, during the AC measurement, a significant effect was observed in two interaction terms involving concentration. During the FRAP measurement, the interaction between microwave power and solvent concentration (BD) was significant, while during the DPPH measurement, the interaction between time and concentration (AD) was significant. Among the quadratic terms, time^2 (A^2) and concentration^2 (D^2) had a significant effect on the responses obtained during extraction.

Figure 3 shows a graphical representation of the models. The solvent concentration was fixed at 2 mM in all plots. Based on the diagrams, microwave power and extraction time had a significant effect on the measured responses. The diagrams of the TPC and TMC clearly illustrate linear systems. On the other hand, the 3D diagrams for AC(FRAP), TFC and AC(DPPH) exhibited similar patterns to those observed in the ethanol solvent. These

parameters increased with microwave power and extraction time up to an optimal level. Following this, a slight decline or plateau was noted.

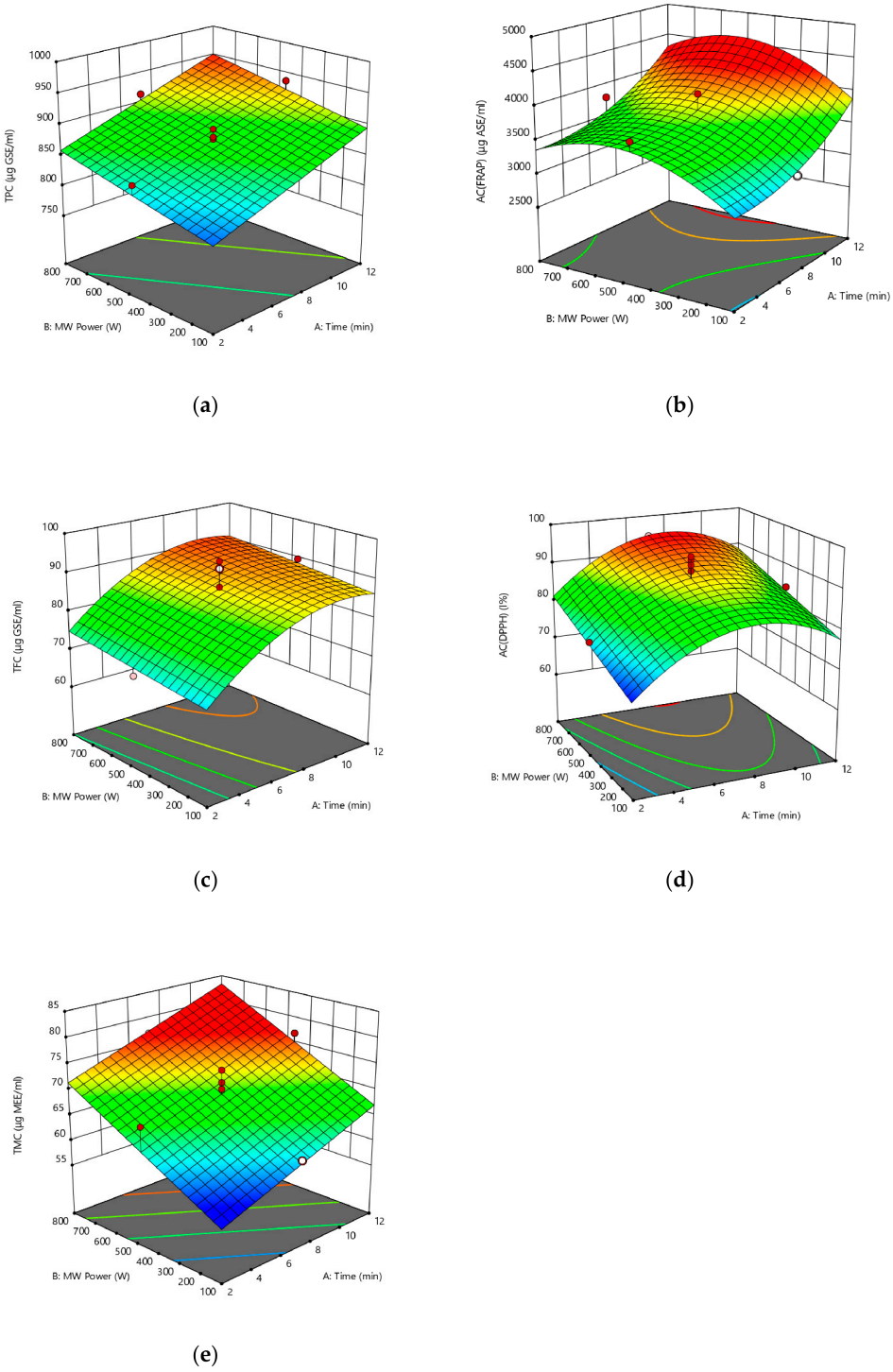


Figure 3. For the β-cyclodextrin-based peppermint extracts, 3D diagrams showing the effect of the independent (process) variables on the responses considered, with the solvent concentration set at medium (2 mM): (**a**) the effect on AC(FRAP), (**b**) the effect on TPC, (**c**) the effect on TFC, (**d**) the effect on AC(DPPH) and (**e**) the effect on TMC.

The FRAP assay revealed that the AC of the β-cyclodextrin-based peppermint extracts varied between 2675.61 ± 262.30 and 5021.02 ± 386.21 µg ASE/mL. In the case of the DPPH assay, the values ranged from 45.78 ± 2.96 to 99.87 ± 3.30%, while the TPC, TFC and TMC were found to range between 682.99 ± 21.72 and 985.92 ± 3.18 µg

GAE/mL, between 57.83 ± 3.35 and 93.76 ± 1.16 µg QUE/mL, and from 56.24 ± 3.22 to 76.88 ± 10.03 µg MEE/mL.

The maximum recovery of each bioactive compound and the corresponding extraction parameters were evaluated individually, which Table 7 shows.

Table 7. For the β-cyclodextrin-based peppermint extracts, the maximum yields for each were response-considered, and the optimal conditions were predicted by desirability.

		Optimized Response				
		AC(FRAP)	**TPC**	**TFC**	**AC(DPPH)**	**TMC**
Conditions Optimal	Time (min)	12.00	12.00	10.21	7.38	10.48
	MW Power (W)	450.00	800.00	800.00	728.417	798.00
	Solvent concentration (mM)	2.00	3.00	1.95	1.18	1.73
Maximum Predicted Value	AC(FRAP) (µg ASE/mL)	4583.79	4318.18	3985.16	3714.22	4033.51
	TPC (µg GAE/mL)	923.05	951.77	934.54	902.12	936.79
	TFC (µg QUE/mL)	88.60	86.28	91.66	86.73	91.43
	AC(DPPH) (%)	83.20	87.05	93.27	95.32	93.09
	TMC (µg MEE/mL)	75.17	76.91	81.46	81.36	83.15

The optimal conditions for extraction were identified as follows: an extraction time of 11.81 min, a microwave power of 800.00 W, and a β-cyclodextrin concentration of 1.70 mM. These parameters were found to maximise the recovery of bioactive compounds. In the context of these optimised conditions, the predicted responses were 949.29 µg GAE/mL for TPC, 90.75 µg QUE/mL for TFC, 4275.54 µg ASE/mL for AC(FRAP), 89.81% for AC(DPPH) and 84.99 µg MEE/mL for TMC.

3.3. The Efficiency of Ethanol Solvents and β-Cyclodextrin Solvents

The extractability of specific bioactive compounds from peppermint using ethanol and β-cyclodextrin solvents are compared in Table 8. The operation conditions were microwave power (100 W, 450 W, and 800 W), extraction time (2 min, 7 min and 12 min) and solvent concentration (ethanol: 10% *v/v*, 35% *v/v* and 60% *v/v*; β-cyclodextrin: 1 mM, 2 mM and 3 mM) (see Tables S1 and S2).

Based on the results, the concentration of β-cyclodextrin was not a statistically significant difference on most responses except for AC(FRAP). The measured values were similar to or higher than those obtained with ethanol. This indicates that the use of β-cyclodextrin had a positive effect on the extraction, and even at low concentrations it increased the extraction efficiency compared to ethanol. A study by Athanasiadis et al. [21] also supports this finding, showing that the concentration of β-cyclodextrin increases beyond a certain limit and had no significant effect on polyphenol extraction, but it produced extracts with a much richer polyphenol composition and better antioxidant properties. In a study conducted by Vhangani et al. [26], no statistically significant differences were observed between the extraction of Green Rooibos using 7.5 and 15 mM of β-cyclodextrin. However, a significant increase in extraction efficiency was observed for pure water extracts.

Table 8. A comparison of the extractability of bioactive compounds from peppermint leaves using ethanol and β-cyclodextrin solvents under MAE.

Specific Compounds	Level	Ethanol	β-Cyclodextrin
AC(FRAP) (μg ASE/mL)	−1	3816.34 ± 324.41 ^{a}	4281.49 ± 340.05 ^{A}
	0	3829.88 ± 32.87 ^{a}	5021.02 ± 386.21 ^{B}
	1	3247.78 ± 88.27 ^{b}	4208.67 ± 385.40 ^{A}
TPC (μg GAE/mL)	−1	1023.85 ± 20.21 ^{a}	917.44 ± 22.47 ^{A}
	0	1037.53 ± 24.64 ^{a}	985.92 ± 3.18 ^{A}
	1	933.61 ± 15.39 ^{b}	956.12 ± 6.09 ^{AB}
TFC (μg QUE/mL)	−1	71.46 ± 1.48 ^{a}	85.87 ± 2.69 ^{A}
	0	68.19 ± 1.93 ^{a}	93.76 ± 1.16 ^{B}
	1	62.26 ± 2.45 ^{b}	86.84 ± 1.35 ^{A}
AC(DPPH) (%)	−1	75.16 ± 0.57 ^{a}	85.00 ± 7.02 ^{A}
	0	73.22 ± 1.60 ^{a}	94.78 ± 3.68 ^{B}
	1	68.10 ± 1.85 ^{ab}	99.86 ± 6.60 ^{B}
TMC (μg MEE/mL)	−1	69.89 ± 9.03 ^{ab}	81.93 ± 6.60 ^{A}
	0	53.78 ± 8.53 ^{a}	83.47 ± 10.89 ^{A}
	1	75.49 ± 14.71 ^{b}	78.43 ± 3.52 ^{A}

Different superscripts indicate significant differences. Lowercase letters (a, b) are used to compare ethanol with the same parameters ($p < 0.05$). Uppercase letters (A, B) are used to compare β-cyclodextrin effects with the same parameters ($p < 0.05$).

Compared to ethanol, the β-cyclodextrin extract showed a 31.5% higher AC(FRAP) antioxidant capacity, 31.2% higher TFC, 32.9% higher AC(DPPH) and 10.6% higher TMC. Only the TPC showed a slight decrease of about 5.0% compared to ethanol. The findings indicate that β-cyclodextrin exhibited superior efficacy in the extraction and stabilisation of bioactive compounds and menthol, while ethanol remained slightly more effective for total phenolics. This was attributed to the inclusion complex formation between β-cyclodextrin and organic compounds, which improved their solubility and thermal stability [30,31]. Gao et al. [53] observed a similar trend in their study of Polygonum cuspidatum, according to which the extraction efficiency of polar components was enhanced by β-cyclodextrin through the formation of by-products, while the extraction of less polar compounds was facilitated by ethanol, in accordance with the "like dissolves like" principle.

In the ethanol solvent system, extraction efficiency did not increase with higher ethanol concentrations. Similar findings have been reported in several studies, indicating that although ethanol enhances the solubility of phenolic compounds, the presence of water is essential [54,55]. Highly concentrated ethanol solutions decrease the extraction of polar compounds by limiting solvent penetration into the plant tissue. The presence of an appropriate amount of water in the solvent can enhance the swelling of the plant matrix, increasing the matrix's contact surface area with the solvent [17,20].

In conclusion, both ethanol and β-cyclodextrin were effective solvents for microwave extraction of peppermint leaves. For ethanol, the lowest concentration (10% *v/v*) was the most effective, which facilitated the recovery of total phenolics. For β-cyclodextrin, a concentration of approximately 2 mM was found to be the optimal choice, which enhanced both the antioxidant activity and the stabilisation of heat-sensitive compounds, such as menthol. Based on the results, β-cyclodextrin could be a suitable green alternative solvent. From a practical perspective, however, the residual ethanol content of extracts must be considered in downstream applications. Processes such as spray drying, vacuum drying, fluidized bed drying and wet granulation may require additional steps, which may be required to remove the ethanol, which increases processing complexity and costs. In contrast, β-cyclodextrin does not present such limitations. Furthermore, it can be used as a

wall material during microencapsulation, providing a dual functional role as an extraction medium and a stabilising carrier.

In future research, it would be worthwhile to investigate the combination of ethanol and β-cyclodextrin as solvents or other alternative green solvents to further enhance extraction efficiency. However, in this case, it must be taken into account that the high ethanol content would significantly reduce the solubility of β-cyclodextrin and impair its complexing properties [56]. The optima (time and MW power) of extraction with a β-cyclodextrin solvent were close to the limit of the tested range; therefore, it would be worthwhile to investigate this in an even larger range in the future in order to increase efficiency.

4. Conclusions

In this study, the extraction of peppermint (*Mentha piperita* L.) by microwave-assisted extraction (MAE) was investigated using ethanol and β-cyclodextrin solvents. For the ethanol solvent, the optimal parameters (in the tested range) were 571 W of microwave power, 7.3 min of extraction time, and 10% v/v solvent concentrations. For the β-cyclodextrin solvent, the optimal parameters (in the tested range) were 800 W of microwave power, 11.81 min of extraction time, and 1.70 mM of solvent concentration. These extraction parameters ensured the maximum yield of phenolics, flavonoids, antioxidants, and menthol.

The β-cyclodextrin solvent system provided similar or higher antioxidant activity and menthol content compared to ethanol, demonstrating its effectiveness in extracting thermolabile bioactive compounds. Although ethanol produced a slightly higher total phenolic content, β-cyclodextrin proved to be a more environmentally friendly alternative, maintaining high extraction efficiency while improving the extraction limit.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/suschem7020024/s1, Table S1: Experimental scheme and results of ethanol extracts; Table S2: Experimental scheme and results of β-cyclodextrin extracts.

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Abbreviations

The following abbreviations are used in this manuscript:

MAE	Microwave-assisted extraction
RSM	Response surface methodology
TPC	Total polyphenol content
TFC	Total flavonoid content
AC	Antioxidant capacity

TMC	Total menthol content
FRAP	Ferric reducing antioxidant power
DPPH	2,2-diphenyl-1-picrylhydrazyl
GAE	Gallic acid equivalents
QUE	Quercetin equivalents
ASE	Ascorbic acid equivalents
MEE	Menthol equivalents
ANOVA	Analysis of variance
SS	Sum of squares

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