



# Article Response Surface Methodology as a Tool for Optimization of Pulsed Electric Field Pretreatment and Microwave-Convective Drying of Apple

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**Abstract:** The benefits of using hybrid drying are increasingly remarked. Microwave-convective drying (MW-CD) links the advantages of both microwave and convective drying methods and allows the negative phenomena that appear when the methods are used separately to diminish. Most importantly, reduced specific energy consumption and relatively short drying time are observed, which can be additionally decreased by the application of various preliminary treatments, e.g., pulsed electric field (PEF). Thus, the purpose of this study was to determine the impact of PEF pretreatment on the MW-CD of apples and its chosen physicochemical properties. This research was designed using response surface methodology (RSM). The first variable was microwave power (100, 200, and 300 W), and the second was specific energy input (1, 3.5, and 6 kJ/kg). Optimization responses were assumed: drying time to MR = 0.02, water activity, hygroscopicity after 72 h, rehydration ratio, relative dry matter content, total phenolic content, ability to scavenge ABTS<sup>•+</sup> radical cations, and DPPH<sup>•</sup> radicals based on the *EC*<sub>50</sub> values. The most optimal parameters were comprised of specific energy intake of 3.437 kJ/kg and microwave power of 300 W (desirability equalled 0.624), which provided the most minimized drying time and obtaining of apples with the most desired properties.

**Keywords:** PEF; microwave-convective drying; hybrid drying; hygroscopicity; rehydration; colour; polyphenols; antioxidant activity; RSM

# 1. Introduction

Drying is a commonly used process in the food industry, the main goal of which is to extend the shelf-life of food by removing some of the water from it [1–3]. In practice, the course of this process as well as the quality of the obtained dried material are strictly dependent on the method used [4,5]. Hybrid drying is increasingly used to improve energy and drying efficiency. It involves combination of at least two methods of drying during a single operation or applying them sequentially—directly one method after another. The synergistic effect of several drying mechanisms, with appropriately selected process parameters, allows to reduce drying time, and thus improve the quality of the dried material by limiting colour changes, preserving smell or flavour, minimizing microstructural changes, as well as increasing the retention of bioactive ingredients [6].

Microwave-convective drying (MW-CD) is an example of hybrid drying. It combines the advantages of convective and microwave drying while minimizing the disadvantages characteristic for these drying methods [5]. Convective drying is based on the removal of water from the material, most often with hot air. The uncomplicated mechanism and relatively low costs make it one of the most frequently used drying methods in the food



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). industry. Significant energy consumption of this method results from the low drying rate. Additionally, due to the use of hot air as a drying agent and relatively long process duration, the quality of the obtained materials is unsatisfactory [7,8]. The use of microwave (electromagnetic waves of specific wavelength) enables quick, volumetric generation of heat (in the entire volume of the material), which causes an increase of internal pressure and pushes the water towards the surface, from where it evaporates intensively. This ensures fast drying of food at low energy consumption. One of the disadvantages of microwave drying is the risk of uneven heating. Difficulties related to controlling the temperature and the rate of drying cause this method to be most often combined with other drying methods, to prevent the risk of damaging the material [5,6,9–11].

In the case of MW-CD, two parameters are considered the most important—microwave power and drying air temperature. Research carried out on onions [12] showed that the assistance of hot air tunnel drying by microwaves (MW-HA) led to a reduction of the drying time by more than 90% (depending on the parameters used), and thus, a decrease of energy consumption by 3–16 times (in comparison to only hot air tunnel drying—HA). In addition, the higher the microwave power and air temperature were used, the higher the drying rate and the shorter the drying time were achieved. This tendency was also observed in the studies carried out on apples [13] and nectarines [14]. Reduced time of convective drying, caused by assisting the process with microwaves, has also been noticed in the studies conducted on durian chips [15], cherry tomatoes [16], pumpkins [17], and shiitake mushrooms [18]. Onions dried by MW-HA exhibited better rehydration properties, flavour, and colour, and also higher retention of total phenolic content and antioxidants than the ones dried with hot-air (HA) [12]. In the case of durian chips, the materials obtained by MW-HA had higher crispness and lightness, but lower hardness and shrinkage compared to HA dried samples. Additionally, a reduced energy consumption of the MW-HA method was observed, in comparison with HA method [15]. Higher retention of total phenolic compounds (including flavonoids), ascorbic acid, and lycopene, as well as better colour and texture preservation, were observed in cherry tomatoes dried by microwave-assisted hot air drying in comparison with hot air drying [16].

Pulsed electric field (PEF) is one of the nonthermal food processing techniques. The essence of its performance lies in the phenomenon of electroporation, i.e., perforation of the material's cell membrane. The cell membrane continuity is broken due to the formation of numerous pores within it [4,19]. Its consequence is an increase of permeability of the cell membrane, which improves the efficiency of many processes, such as osmotic dehydration [20,21], freezing [22], extraction [23], or drying [24,25].

The majority of the studies in the literature concern the impact of pulsed electric field pretreatment on the subsequent convective drying [24,26–29], less frequently vacuum drying [30–32], and freeze-drying [25,33]. Only a few articles present results on the effect of this nonthermal pretreatment on the subsequent microwave-convective drying.

So far, the impact of the pulsed electric field on microwave-convective drying has been mainly investigated in the case of vegetables, i.e., carrots [34,35] and potatoes [36–39]. The literature also provides information on the impact of PEF pretreatment on the chemical properties (total phenolic compounds and antioxidant activity) of apples dried with the use of microwave and hot air. The use of this nonthermal pretreatment led to a reduction of microwave-convective drying time of carrots and apples, by 38% and 27%, respectively [34], as well as potatoes by 23% [36]. Moreover, the application of PEF before microwave-convective drying of carrots increased the carotenoid retention by 20%, however it did not significantly affect the antioxidant activity and total phenolic compounds content in the obtained dried apples (in reference to the samples untreated with PEF before drying). The electroporation of the tissues of these plant materials caused an increase in the mobility of water molecules, which, by accelerating the drying process, reduced the oxidative and thermal degradation of nutrients contained in these materials [34].

As revealed, the microwave-convective drying process of fruits with PEF pretreatment has not been analysed in great detail so far. Apart from the partially studied impact of PEF pretreatment on the chemical properties of some microwave-convective dried plant materials, there is no information about the impact of the electric field on the physical properties of materials dried with this method (i.e., colour, hygroscopicity, or rehydration). These properties are particularly important for dried products and determine their quality [5]. Therefore, conducting further research in this area seems justified.

This study aimed to determine the effect of pulsed electric field pretreatment on the microwave-convective drying of apples and the selected physicochemical properties of the obtained dried materials. Furthermore, the most optimal parameters of PEF pretreatment (specific energy intake) and microwave-convective drying (power of microwave) were determined, which are essential for minimizing the drying time of apples and obtaining dried material with the desired properties.

# 2. Materials and Methods

## 2.1. Material

The 'Golden Delicious' variant of apples was chosen for the material of the study, which was acquired from the Experimental Fields of the Department of Fruit Growing of the Warsaw University of Life Sciences (SGGW, Warsaw, Poland). Preceding the experiment, the apples were kept in cold storage ( $5 \pm 1$  °C) for a period of one week. In preparation for the experiment, collected apples were subjected to a selection process, and based on their shape, colour, and maturity the most standard specimens were chosen for the tests. Before initiating technological processes, apples were taken out of the cold storage and were allowed to attain room temperature (about 20 °C), after which they were thoroughly washed with tap water ( $21 \pm 1$  °C). The dry matter content in raw material (fresh apple) equalled 14.5  $\pm$  0.1%.

The dried materials were stored for one week at room temperature ( $20 \pm 1 \degree C$ ) after tightly packing them (directly after microwave-convective drying) into light barrier pouches impermeable to gas and vapour, made of laminate (PET/AI/PE).

#### 2.2. Design of Experiments—Response Surface Methodology (RSM)

The evaluation of the impact that PEF pretreatment and microwave-convective drying parameters have on selected dependent variables was performed utilizing the central composite design (face-centered, CCF). The design was also used to assess the possible optimization of both processes. Two variable factors were chosen for the study, each differentiated by three levels. Microwave power (factor 1) was differentiated with the following levels: 100, 200, and 300 W. Specific energy intake (factor 2) was set at 1, 3.5, and 6 kJ/kg. The parameters of PEF treatment and drying conditions were selected based on the results of preliminary experiments. In essence, the study was arranged into performing 11 experiments corresponding to a specific combination of each factor at each level, with triple repetition conducted at the center point (0, 0). The actual values of parameters are presented in Table 1. To regularize current values of factors, they were subjected to normalization by attributing coded values to them. These coded values ranged from 1 to -1and were assigned in the following manner: the highest level of the factor was appointed with 1, the medium level of the factor was appointed with 0, and finally the lowest level of the factor with -1. The following parameters were used as responses: drying time to MR = 0.02, water activity, hygroscopicity after 72 h, rehydration ratio, relative dry matter content, total phenolic content, ability to scavenge ABTS<sup>++</sup> radical cations and DPPH<sup>+</sup> radicals based on the  $EC_{50}$  values. Moreover, reference processes, carried out without any pretreatment at three different levels of microwave power, were performed for comparison purposes.

	Fac	tor 1	Factor 2		
Run	Microwave Power [W]	Coded Value	Specific Energy Intake [kJ/kg]	Coded Value	
1	200	0	3.5	0	
2	300	1	6	1	
3	200	0	6	1	
4	300	1	1	-1	
5	300	1	3.5	0	
6	200	0	3.5	0	
7	100	-1	3.5	0	
8	100	-1	6	1	
9	200	0	1	-1	
10	200	0	3.5	0	
11	100	-1	1	-1	

## 2.3. Pulsed Electric Field Pretreatment

The PEF pretreatment of apples was performed utilising a batch system (PEFPilot<sup>TM</sup> Dual System, Elea Vertriebs- und Vermarktungsgesellschaft mbH, Quakenbrück, Germany). To accomplish that, in the beginning, a single apple was placed within a chamber, the capacity of which equalled 2-L. The build of the chamber included a set of two parallel stainless-steel electrodes, which were distanced from each other by 24 cm. Once the apple was in place, the treatment chamber was filled with water ( $21 \pm 1$  °C), which served the function of a conductive medium. When weighed, the total input of the chamber added up to approximately 1.5 kg, consistent for each experiment. The parameters of the pulsed electric field were set at the following values: electrode voltage equalled 24 kV, electric field strength stood at 1 kV/cm, pulse frequency was at 20 Hz, while the pulse width took the value of 7  $\mu$ s. The number of rectangular pulses varied concerning the amount of supplied energy (1, 3.5, 6 kJ/kg).

The values of the specific energy intake  $W_{spec}$  [kJ/kg] and electric field strength *E* [kV/cm] were calculated in accordance with the following formulas:

$$W_{spec} = \frac{I \cdot U \cdot t \cdot n}{1000 \cdot m} \tag{1}$$

$$E = \frac{U}{d} \tag{2}$$

where: *I*—current [A]; *U*—voltage [V]; *t*—duration of a pulse [s]; *n*—number of pulses [-]; *m*—mass of the total input [kg]; *d*—distance between electrodes [cm].

Using a conductometer (pH/conductivity meter CPC-401, Elmetron, Zabrze, Poland) for determining apples' conductivity before and after the PEF treatment allowed for calculation of the CDI (cell disintegration index), which was executed in accordance with the literature [27]:

$$CDI = \frac{\sigma - \sigma_i}{\sigma_d - \sigma_i} \tag{3}$$

where:  $\sigma$ —conductivity of tissue after applying a pulsed electric field [ $\mu$ S];  $\sigma_i$ —conductivity of intact tissue [ $\mu$ S];  $\sigma_d$ —conductivity of maximally ruptured tissue [ $\mu$ S].

#### 2.4. Microwave-Convective Drying

The microwave-convective drying process was carried out on quarters of apple slices. These apples underwent prior preparation, which consisted of firstly cutting the fruits into slices (5 mm thick), then removing their cores, and finally cutting the slices into four equal pieces.

The procedure itself, regarding both untreated and PEF-treated apples, was performed in a laboratory dryer (Promis-Tech Inc., Wroclaw, Poland). The majority of conditions were invariant throughout the drying process, as the air temperature was fixed at 30 °C, while the air velocity was set at 2 m/s, which was maintained for all variants. At the same time, the microwave frequency equalled 2.45 GHz. Only one of the factors was variable, as the microwave power took values of 100, 200, and 300 W. Once prepared, apples were arranged in a single layer on a rotating cylindrical sieve, a load of which equalled  $3.75 \text{ kg/m}^2$ . During the course of drying, the weight of apples was being recorded in 5 min intervals, which was aimed at determining the drying time (time necessary to achieve MR = 0.02 by dried apples). Each time the drying process was carried out until stable, unchanged weight measurements were achieved for a period of 15 min. Calculation of MR (moisture ratio) was done in accordance with the literature [40]:

$$MR = \frac{M_{\tau}}{M_0} \tag{4}$$

where:  $M_{\tau}$ —water content in samples during drying [kg H<sub>2</sub>O/kg d.m.];  $M_0$ —initial water content in samples [kg H<sub>2</sub>O/kg d.m.].

The drying process was performed thrice.

#### 2.5. Dry Matter Content

Evaluation of the dry matter content (d.m.), respectively, for fresh and dried material, was carried out via gravimetric method in accordance with the AOAC 920.15, 2002 standard [41] thrice.

#### 2.6. Water Activity

The water activity assessment was conducted with the usage of a hygrometer (AquaLab CX-2, Decagon Devices, Pullman, WA, USA), threefold, at which time the temperature was at room level (about 20 °C).

#### 2.7. Hygroscopic Properties

Assessment of hygroscopic properties was focused on water vapour adsorption capacity ( $H_{72h}$ ) of the obtained dried product. This was achieved through a modified method described in the study [42]. Adsorption of water vapour lasted 72 h and during this process the analysed samples (weighed and put in the aluminium dishes) were placed over a saturated NaCl solution with water activity equal to 0.75, at room temperature (approx. 20 °C). The results of the analysis, which was performed thrice, were expressed as ratio stated below:

$$H_{72h} = \frac{m_{\tau}}{m_0}$$
(5)

where:  $m_{\tau}$ —the mass of the dried apples after adsorption process [g];  $m_0$ —the initial mass of the samples [g].

#### 2.8. Rehydration Properties

The dried apples were examined concerning their rehydration properties. For that purpose, a modified method developed by [43] was used. The rehydration process was performed using distilled water at the temperature of  $20 \pm 1$  °C, in which analysed samples were immersed for 30 min (the ratio between the mass of dried material compared to the mass of water equalled 1:100). After completion of the process, rehydrated material was subsequently strained, blotted with the use of filter paper, and weighed once more. The analysis was carried out in three consecutive repetitions. Subsequently, obtained results allowed for calculation of the rehydration ratio (RR) as well as the relative dry matter content (SSL) as stated hereafter:

$$RR = \frac{m_{\tau}}{m_0} \tag{6}$$

$$SSL = \frac{m_{\tau} \cdot d.m_{\cdot\tau}}{m_0 \cdot d.m_{\cdot 0}} \tag{7}$$

where:  $m_{\tau}$ —the mass of the sample after subjecting it to rehydration [g];  $m_0$ —the mass of the sample prior to rehydration [g];  $d.m._{\tau}$ —dry matter content in the sample after subjecting it to rehydration [%];  $d.m._0$ —dry matter content in the sample prior to rehydration [%].

## 2.9. Colour

Both fresh and dried products were examined in terms of colour characteristics via a trichromatic colorimeter (CR-5, Konica-Minolta, Tokyo, Japan). For that purpose, an analysis was carried out utilizing reflected light and the CIE L\*a\*b\* system (standard observer was set at  $2^{\circ}$ , D65 was used as a standard illuminant, while the diameter equalled 3 mm). The described analysis was iterated ten times. Upon receiving specific values of the L\*, a\*, and b\* colour parameters that were registered during the analysis, the total colour difference ( $\Delta E$ ) was calculated as shown below:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$
(8)

where  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  stand for the differences between the values of the L<sup>\*</sup>, a<sup>\*</sup>, b<sup>\*</sup> of dried and fresh apples.

## 2.10. Preparation of the Extracts

The dried material was ground with the usage of an analytical mill (A11 basic, IKA<sup>®</sup>-Werke GmbH & Co. KG, Staufen, Germany). After carrying out that process, exactly 1 g of the obtained powder was placed inside of a glass beaker with 20 mL of 80% ethanol solution. Afterwards, the prepared solution was heated moderately for 3 min, however maintained below the boiling point. It was then subsequently filtered and filled up to the level of 50 mL. With the help of these extracts, total phenolic content and antioxidant activity were evaluated. For each of the samples, there was an extract composed in duplicate.

#### 2.11. Total Phenolic Content

As for the total phenolic content (TPC) present in obtained dried apples, it was calculated in alignment with Folin–Ciocalteu's method [44], in which case gallic acid was utilized as a standard. The contents of a glass tube, which included 4.92 mL of distilled water, 0.18 mL of apple extract, and 0.3 mL of Folin–Ciocalteu reagent, were mixed. After 3 min, another ingredient, specifically 0.6 mL of supersaturated sodium carbonate solution was added to the mixture and subjected to mixing once more. The incubation process was conducted in the darkness for the period of 60 min at room temperature (approximately  $20 \,^{\circ}$ C). The solutions developed to assess absorbance of the extracts were measured with the use of a spectrophotometer (He $\lambda$ ios Thermo Electron v. 7.03, Thermo Electron Corporation, Waltham, MA, USA) set at a wavelength of 750 nm, in reference to a blank sample (without extract, but with 0.18 mL of 80% ethanol solution). Said analysis was conducted in four repetitions (twice for each of the extracts) and the obtained results were defined as mg of gallic acid (GAE) per 100 g of dry matter.

## 2.12. Antioxidant Activity (ABTS and DPPH Assays)

The core of the analysis was the assessment of the scavenging degree of ABTS<sup>•+</sup> radical cations and DPPH<sup>•</sup> radicals [45] which was caused by antioxidants that were within the used extracts. The preparation of the free radical solutions of ABTS<sup>•+</sup> and DPPH<sup>•</sup>, as well as the solutions for the measurements, are described in the study [46]. Glass tubes were filled with 0.025, 0.050, 0.075, 0.100 mL, and 0.1, 0.2, 0.3, 0.4 mL of apple extracts for ABTS and DPPH assays, respectively. 80% ethanol solution was added to each glass tube intended for the DPPH assay, in such an amount that the total volume of the glass tube equalled 2 mL. Afterwards, the free radical solutions were dispensed into glass tubes in volumes of 3 mL and 2 mL for ABTS and DPPH assays, respectively. The prepared solutions were mixed and allowed to incubate for 6 min (ABTS assay) and 30 min (DPPH assay) at room temperature (approximately 20 °C), in darkness. The created solutions

were examined in terms of absorbance, for which a spectrophotometer was put to use (He $\lambda$ ios Thermo Electron v. 7.03, Thermo Electron Corporation, Waltham, MA, USA) at two different wavelengths, specifically 734 nm (ABTS assay), and 515 nm (DPPH assay). This evaluation was executed in four repetitions to examine each of the extracts twice, after which the observed results were converted into  $EC_{50}$  coefficient, which stands for an extract concentration necessary for a 50% reduction of the initial amount of ABTS<sup>++</sup> radical cations and DPPH<sup>•</sup> radicals. Regarding the mentioned coefficient, it was calculated as mg of dry matter per 1 mL of extract.

#### 2.13. Statistical Analysis

The obtained results (dry matter content and total colour difference) were put through statistical analyses: one-way analysis of variance ANOVA ( $\alpha = 0.05$ ) and post hoc analysis consisting in Tukey's test (Statistica v. 13.3, TIBCO Software Inc., Palo Alto, CA, USA), to specify statistical differences between them.

The execution of RSM modeling was carried out through the utility of Design-Expert software (v. 13.0.9.0, Stat-Ease Inc., Minneapolis, MN, USA). Obtained results underwent statistical verification using results from ANOVA. The model terms were concluded to be significant based on *p*-values < 0.05. The developed model was verified in terms of suitability based on obtained parameters, including  $R^2$  (determination coefficient), CV (coefficient of variation), *p*-value, and lack of fit test.

#### 3. Results and Discussion

## 3.1. Cell Disintegration Index

The cell disintegration index (CDI) was used to determine the structural changes induced by electroporation of the cell membrane due to the action of a pulsed electric field. This index takes values between 0 and 1—CDI = 0 indicates intact cells, while CDI = 1 stands for totally disintegrated cells of the material tissue [27]. Undoubtedly, the values of this index are influenced by the parameters used, such as temperature, the number of pulses supplied, or the intensity of the electric field, but the structure of the treated material is also important [4,24,47]. The more energy was supplied to the apples during the PEF treatment, the more the tissue of this plant material was damaged (Table 2). This tendency is consistent with the results obtained by other authors [26,33]. The supply of a variable amount of energy (1–6 kJ/kg), with the electric field strength E = 1 kV/cm, resulted in the CDI in the range of 0.08–0.37.

**Table 2.** Values of CDI (cell disintegration index), dry matter content (d.m.), and  $\Delta E$  (total colour difference) of obtained dried apples.

Microwave Power [W]	Specific Energy Intake [kJ/kg]	Sample Code	CDI [-]	d.m. [%]	ΔE [-]
100	0	MW-CD100	-	$92.5\pm0.0~^{\rm b}$	$12.8\pm3.2~^{\mathrm{abc}}$
	1	MW-CD100_PEF1	0.09	$92.6\pm0.0$ <sup>b</sup>	$14.2\pm3.0~\mathrm{bcd}$
	3.5	MW-CD100_PEF3.5	0.23	$92.4\pm0.0$ <sup>b</sup>	$11.8\pm2.6~^{ m abc}$
	6	MW-CD100_PEF6	0.36	$90.7\pm0.1$ a	$14.2\pm1.8~^{bcd}$
200	0	MW-CD200	-	$94.4\pm0.1~^{ m cd}$	$13.7\pm4.0~^{ m abcd}$
	1	MW-CD200_PEF1	0.09	$94.8\pm0.2$ <sup>de</sup>	$11.6\pm2.9$ $^{ab}$
	3.5	MW-CD200_PEF3.5	0.27	$94.4\pm0.2$ <sup>c</sup>	$13.7\pm3.3~\mathrm{bc}$
	6	MW-CD200_PEF6	0.36	$94.9\pm0.1~^{e}$	$16.1\pm3.1~^{\mathrm{bcd}}$
300	0	MW-CD300	-	$96.5\pm0.1~^{\rm f}$	$16.5\pm4.2~^{\mathrm{cd}}$
	1	MW-CD300_PEF1	0.08	$96.4\pm0.1~^{ m f}$	$9.0\pm2.4$ a
	3.5	MW-CD300_PEF3.5	0.21	$96.5\pm0.0$ f	$15.9\pm4.3~\mathrm{bcd}$
	6	MW-CD300_PEF6	0.37	$97.1\pm0.0~{ m g}$	$17.9\pm3.8$ <sup>d</sup>

The results are presented as the mean  $\pm$  standard deviation, and different letters in the columns indicate significant differences between the results (p < 0.05).

## 3.2. Drying Kinetics and Drying Times

Figure 1 presents the drying curves of untreated and PEF-treated apples dried by the microwave-convective method. At the beginning of the process, a significant efficiency of water removal was observed, which was related to the evaporation of easily available capillary and surface water [48]. As drying progressed, the efficiency of the process decreased due to increasing difficulties in removing water more bound to the material [49]. As can be seen, the course of microwave-convective drying of apples differed depending on the amount of energy supplied during the PEF treatment and the level of microwave power. The drying time of the apples untreated with PEF and dried at a microwave power of 100, 200, and 300 W equalled  $200 \pm 0$ ,  $110 \pm 0$ , and  $60 \pm 0$  min, respectively. The quadratic model allowed the significant prediction (p < 0.05) of the drying time of apples treated with PEF and dried using the microwave-convective method. Moreover, a very good fit of the model to the data obtained experimentally was observed, which was confirmed by the high value of the determination coefficient ( $R^2 = 0.9846$ ) and the insignificant result of the lack of fit test (p > 0.05) presented in Table 3. ANOVA showed that the drying time of the tested samples was significantly influenced (p < 0.05) only by microwave power (linear and quadratic effects). Based on Figure 2, it can be seen that the drying time decreased with increasing the power of the microwave. The obtained tendency is consistent with the observations of other authors [50–52]. The drying time of apples subjected to PEF was considerably reduced (by 8-28%) compared to untreated samples. However, no significant effect of the amount of energy supplied during the pretreatment (1-6 kJ/kg) on this parameter was found (p > 0.05). Such results suggest that application of lower specific energy input during PEF treatment may be justified, taking into account energy and environmental factors. Reduced drying time generally results in a decrease of energy consumption [40,53–55], so the application of PEF, by intensifying the MW-CD of apples, probably led to a reduction in the amount of energy used. Based on Equation (9), where the factors should be specified in actual values, it is possible to calculate the drying time of PEF-treated and microwave-convective dried apples. The use of PEF pretreatment resulted in damage to the apple tissue (according to CDI) at the level of 0.1–0.4 (Table 2). This means that this process, to some extent, led to a break in the continuity of the cell membrane [46]. The pores in the cell membrane created as a result of electroporation lead to an increase in the mobility of various molecules, thanks to which the effectiveness of diffusion-based processes increases, e.g., drying [47,56]. This may explain the achieved reduction in drying time for apples subjected to PEF treatment compared to untreated ones. During microwave drying, the radiation energy is absorbed by the material. The microwave energy is transformed into thermal energy, which heats the material in its entire volume [50,57]. Increasing the microwave power results in an increase in the temperature of the material, which causes a growth in both internal pressure and the concentration gradient. For this reason, the evaporation of water is intensified, which reduces the drying time [51,52].

Drying Time = 276.60526 - 1.33504 · Microwave Power - 7.82456 · Specific Energy Intake +0.005 · Microwave Power · Specific Energy Intake + 0.002211 · Microwave Power<sup>2</sup> +0.736842 · Specific Energy Intake<sup>2</sup>

(9)



Figure 1. Drying curves of microwave-convective drying of apples untreated or PEF-pretreated.

**Table 3.** The results of statistical tests of fitting the equations of the response surfaces to the values obtained experimentally ( $\alpha = 0.05$ ); R<sup>2</sup> (determination coefficient), CV (coefficient of variation).

Response	Model	R <sup>2</sup>	CV [%]	<i>p-</i> Value (Model)	<i>p-</i> Value (Lack of Fit)
Drying Time	Quadratic	0.9846	6.51	0.0002	0.3464
Water Activity	Quadratic	0.9471	4.60	0.0033	0.4502
$H_{72h}$	Quadratic	0.9541	0.4477	0.0023	0.4658
RR	Linear	0.5437	5.43	0.0433	0.3580
SSL	Linear	0.5753	3.55	0.0325	0.2979
TPC	Quadratic	0.8755	15.52	0.0259	0.1358
$EC_{50}$ ABTS	Reduced Quadratic	0.7966	17.02	0.0285	0.4117
$EC_{50}$ DPPH	Reduced Quadratic	0.7608	14.26	0.0449	0.6957

The *p*-Value (model) < 0.05 and *p*-Value (lack of fit) > 0.05 indicate a significant fitting of the model.



**Figure 2.** Response surface of drying time to MR = 0.02 of apples treated with PEF prior to microwaveconvective drying, depending on the microwave power (factor A) and specific energy intake (factor B).

## 3.3. Physicochemical Properties

Water activity is one of the most important parameters in assessing food quality and safety. Sufficiently low water activity limits the growth of microorganisms and reduces

the activity of enzymes [58]. The water activity of the apples untreated with PEF and dried at a microwave power of 100, 200, and 300 W equalled  $0.239 \pm 0.014$ ,  $0.205 \pm 0.014$ , and 0.200  $\pm$  0.002, respectively. With the quadratic model, it is possible to significantly (p < 0.05) predict the values of water activity of apples pretreated with PEF and dried in the microwave-convective dryer. Additionally, based on the high value of the coefficient of determination ( $R^2 = 0.9471$ ) and the insignificant result of the lack of fit test (p > 0.05), a very good fit of the model to empirical data was found (Table 3). Based on the analysis of variance, it can be concluded that the water activity was significantly influenced by the microwave power (linear and quadratic effects), specific energy intake (linear effect), and the interaction between these two factors (p < 0.05). As shown in Figure 3a, in most cases, increasing the microwave power and reducing specific energy intake resulted in a reduction in water activity. The quadratic effect was manifested by a slight increase in the water activity of apples treated with the PEF treatment, during which energy was supplied in the amount of 1 kJ/kg, and then dried with a microwave power of 300 W. The water activity of PEF-treated and microwave-convective dried apples can be determined via Equation (10), in which factors should be expressed in actual values.





The effectiveness of the drying process depends, among others, on the properties of the dried material and the applied process parameters [49]. The dry matter content in all obtained dried apples varied between 90.7% and 97.1% (Table 2), which shows that they contained 2.9–9.3% of water. The achieved dry matter content in dried apples is consistent with the results (88.5–95.0%) obtained by other authors [59–62]. With such a low water content, the water activity in the range of 0.188–0.292 ensured the safety of the obtained dried materials [63]. As it is visible in Table 2, apples dried at the lowest microwave power exhibited the lowest dry matter content. Under such drying conditions (100 W, 30 °C), the drying process was not very intensive, which is confirmed by the shape of the drying curves shown in Figure 1. Therefore, it would have been probably impossible to remove more water from the material dried at such process parameters during a reasonable time. With the growth of microwave power, the diffusivity of water molecules also increases [51,57,64,65]. Using the highest power of microwave (300 W) resulted in obtaining dried apples with the

highest dry matter content. This was due to the increased intensity of water evaporation, thus a significant amount of it was removed from the material.

Based on the hygroscopic properties of dried materials, it is possible to determine their storage stability [66], as well as to assess the structural changes caused by a specific drying method or the applied preliminary treatment [67]. The mass gain  $(H_{72h})$  after 72 h of water vapour adsorption (taking place in an environment with  $a_w = 0.75$ ) of the apples untreated with PEF and dried at a microwave power of 100, 200, and 300 W equalled 1.2079  $\pm$  0.0007,  $1.2357 \pm 0.0020$ , and  $1.2481 \pm 0.0004$ , respectively. The quadratic model allowed us to significantly predict (p < 0.05) the values of this parameter for apples treated with PEF and dried using the microwave-convective method. Moreover, a very good fit of the model to the data obtained experimentally was observed, which was confirmed by the high value of the determination coefficient ( $R^2 = 0.9541$ ) and the insignificant result of the lack of fit test (p > 0.05) presented in Table 3. ANOVA showed that the hygroscopic properties of the tested samples significantly were influenced (p < 0.05) by microwave power (linear and quadratic effects) and specific energy intake (linear effect). Based on Figure 3b, the values of  $H_{72h}$  decreased with increasing the amount of energy supplied during PEF treatment and with reducing the power of microwave from 250 to 100 W (between 300 and 250 W a slight growth of this parameter was observed). Equation (11) allows for quantifying the values of  $H_{72h}$  of PEF-treated and microwave-convective dried apples, provided its factors consist of actual values. The hygroscopic properties depend, among others, on the structure of a particular material, but the more it is damaged, the lower these properties are, which is important from the point of view of the storage stability [67]. According to the calculated CDI values (Table 2), the more energy that was supplied to the apples during the PEF treatment, the more disintegrated the tissue of this material was, which could result in a reduced ability to water vapour adsorption. Additionally, as a result of intense electroporation, changes in sugar distribution could have occurred [68]. In turn, during drying, together with the migration of water, there may be the transport of soluble solids directed towards the surface [69]. Stiffening of the external layer of the material due to drying could be a barrier to the adsorption of water vapour. As can be seen from Figure 1, the lower the microwave power, the longer the drying time of the apples. At lower drying rates, higher material shrinkage [52,70] is observed, which may also negatively affect the hygroscopic properties.

 $Water Activity = 0.272047 - 0.000748 \cdot Microwave Power + 0.007208 \cdot Specific Energy Intake$  $-0.000086 \cdot Microwave Power \cdot Specific Energy Intake + 1.99868 \cdot 10^{-6}$ (10)  $\cdot Microwave Power^2 + 0.002118 \cdot Specific Energy Intake^2$ 

 $H_{72h} = 1.15682 + 0.000812 \cdot Microwave Power - 0.006718 \cdot Specific Energy Intake + 0.000015$  $\cdot Microwave Power \cdot Specific Energy Intake - 1.69403 \cdot 10^{-6} \cdot Microwave Power^2$ (11)

+0.000156.*Specific Energy Intake*<sup>2</sup>

Rehydration can be used to assess the impact of a pretreatment or a specific drying method on the structure of the dried material. This process, in contrast to drying, consists of water uptake by the dried material, which increases its volume and weight [43,52,67,69]. The degree of rehydration depends, among others, on the type of matrix, the structural and chemical changes caused by processing, but also on the time of rehydration [14,52,70]. Basically, during rehydration, the ability of the dried tissue of the material to rebind water is evaluated [43,59]. The rehydration ratio (RR) of the apples untreated with PEF and dried at a microwave power of 100, 200, and 300 W equalled 2.2754  $\pm$  0.0472, 2.3874  $\pm$  0.1133, and 2.3151  $\pm$  0.0357, respectively. Despite the relatively weak fit of the model to empirical data (R<sup>2</sup> = 0.5437), based on the insignificant (*p* > 0.05) result of the lack of fit test (Table 3) and Adeq Precision = 6.7133 (adequate precision test, values > 4 indicate the possibility of using the model), it was found that a linear model could be used to significantly (*p* < 0.05) predict the RR values of apples treated with PEF and then dried by the microwave-convective method. ANOVA showed that only microwave power had a significant (*p* < 0.05) linear

effect on the RR of the tested samples. The RR of PEF-treated apples dried via microwaveconvective method can be determined by applying Equation (12), on the condition that factors within it are described with actual values. The values of RR increased with increasing microwave power (Figure 4a), which is consistent with the literature data [52,57,70]. Despite no significant impact of PEF treatment on this parameter (p > 0.05), an increase in RR was observed with a decrease in specific energy intake. Perhaps the higher amount of energy supplied during the PEF treatment resulted in an overprocessing of the material, making it impossible to fully replicate the initial water content. On the other hand, damage to apples at the level of CDI = 0.1 (specific energy intake: 1 kJ/kg) could lead to optimal porosity, limiting shrinkage of the obtained dried apples and thus an improved ability to absorb water [33,52]. Drying of apples at low microwave power (100 W) lasted the longest (Figure 1), which could lead to the highest shrinkage of the samples and thus a reduction in the water absorption ability. The use of higher microwave power could lead to an increase in the porosity of the material and the rapid creation of a barrier surface layer, which, by preserving the volume of dried apples, could reduce their shrinkage and thus lead to an increase in the RR [52,70].





As a result of immersing the dried material in water during the rehydration process, apart from water absorption and swelling of the material, the leaching of soluble solids undoubtedly occurs [52,67]. The relative dry matter content (SSL) in the apples untreated with PEF and dried at a microwave power of 100, 200, and 300 W equalled  $0.7545 \pm 0.0177$ ,  $0.6956 \pm 0.0327$ , and  $0.7337 \pm 0.0017$ , respectively. Based on the linear model, it is possible to significantly (p < 0.05) predict the SSL values of microwave-convective dried apples with PEF pretreatment. This was confirmed by the insignificant (p > 0.05) result of the lack of fit test (Table 3) and the value of 7.1714 obtained in the adequate precision test. Unfortunately, a relatively weak fit of the selected model to the data obtained experimentally ( $R^2 = 0.5753$ ) was found. According to the results of the analysis of variance, only the microwave power (linear effect) had a significant (p < 0.05) impact on the SSL. To evaluate the SSL of PEF-treated and microwave-convective dried apples Equation (13) can be used, but both factors should be specified in actual values. Figure 4b shows that the SSL values increased with the decrease of the loss of soluble

solids. Despite the lack of a significant effect (p > 0.05), an increase in the value of SSL was observed with increasing the amount of energy supplied during the PEF treatment. Based on the previously described results for the RR parameter, it can be concluded that the lower the amount of energy supplied during PEF treatment and the higher the microwave power used during drying, the higher the ability of dried apples to absorb water (the highest RR values) and the higher the risk of significant leaching of soluble solids (the lowest SSL values).

#### $RR = 2.00682 + 0.001203 \cdot Microwave Power - 0.033101 \cdot Specific Energy Intake$ (12)

#### $SSL = 0.787578 - 0.000296 \cdot Microwave Power + 0.008321 \cdot Specific Energy Intake$ (13)

Using the measured values of the colour parameters:  $L^*$  (58.2–79.7),  $a^*$  (–3.2–8.7), and b\* (18.5–37.3) of dried apples, the total colour difference ( $\Delta E$ ) of the obtained materials was calculated in relation to fresh apple (L\* = 77.4  $\pm$  0.3, a\* =  $-2.9 \pm 0.4$ , b\* = 17.7  $\pm$  1.9). As shown in Table 2, the  $\Delta E$  values ranged from 9.0 to 17.9, which indicates a significant colour deviation, visible even by an untrained observer [71]. Such high  $\Delta E$  values obtained for dried apples could be the result of the methodology used, where the light reflected from a porous surface, devoid of a significant amount of water (dried materials), is compared to the surface containing water (fresh material) [30]. Despite the lack of significant differences (p > 0.05), an increase in the values of the  $\Delta E$  parameter was observed as the microwave power increased during the drying of untreated and PEF-pretreated apples (specific energy intake: 3.5 and 6 kJ/kg). This could be due to the supply of more microwave energy to the samples, thus increasing their temperature. Such conditions conduce not only the caramelization of sugars but also the formation of Maillard reaction products, which could lead to nonenzymatic browning of dried materials [50,51,58]. Additionally, increasing the amount of energy supplied during the PEF pretreatment resulted in a higher, but statistically insignificant (p > 0.05), colour deviation of dried apples (at the microwave power of 200 and 300 W). The calculated CDI values indicated that the more energy was supplied, the more ruptured the apple tissue was (Table 2), which could also mean increased cell membrane permeability. As a result of electroporation, the content of cells could be released, including, for example, substrates for polyphenol oxidase, which could result in enzymatic browning of the analysed samples [21]. With the increase in microwave power at a specific amount of PEF energy supplied equalling 1 kJ/kg, a decrease in the values of  $\Delta E$  was observed, which led to the material (MW-CD300\_PEF1) with the lowest total colour difference (p < 0.05). Perhaps the damage to the apple tissue at the level of CDI = 0.1 ensured the most optimal permeability of the cell membrane, thanks to which both the mass transport during drying and the colour of the obtained dried material were more homogeneous [72]. Moreover, such an amount of specific energy intake could be conducive to partial inactivation of enzymes responsible for enzymatic browning of food [1]. High diversity of the optical properties is also related to the native distribution of enzymatic browning substrates, which is not equal, and the percolation associated with the electroporation.

Drying and other methods of food processing lead to changes in the physical and chemical properties of the treated materials [73]. The total phenolic content (TPC) of the apples untreated with PEF and dried at a microwave power of 100, 200, and 300 W equalled 1003.09  $\pm$  43.30, 1379.26  $\pm$  29.89, and 1649.48  $\pm$  25.04 mg GAE/100 g d.m., respectively. With the quadratic model, it is possible to significantly (p < 0.05) predict the values of TPC of apples pretreated with PEF and dried in the microwave-convective dryer. Additionally, based on the relatively high value of the determination coefficient (R<sup>2</sup> = 0.8755) and the insignificant result of the lack of fit test (p > 0.05), a good fit of the model to empirical data was found (Table 3). Based on the analysis of variance, it can be concluded that the TPC was significantly influenced only by the microwave power with linear and quadratic effects (p < 0.05). One can apply Equation (14) to obtain the specific TPC for PEF-treated and microwave-dried apples, however, its factors must be based on actual values. According to the response surface shown in Figure 5, the TPC values decreased with increasing

microwave power to a level of about 170 W, and from this value, an increase in TPC was observed to reach a maximum with a microwave power of 300 W. Despite no significant impact of PEF treatment on this parameter (p > 0.05), an increase in TPC was observed with a decrease in specific energy intake. Supplying the highest amount of specific energy (6 kJ/kg) resulted in a CDI of approximately 0.4 (Table 2). This degree of damage could lead to a significant increase in the permeability of the cell membrane. As a result of electroporation, various components, including phenolic compounds, could be released from the vacuole together with water during pretreatment [34]. Therefore, they could degrade more easily and faster during drying. Supplying the lowest amount of specific energy (1 kJ/kg) during the PEF pretreatment of apple could promote partial inactivation of enzymes [1], e.g., polyphenol oxidase, thus reducing the risk of oxidation of phenolic compounds [74]. Higher TPC values in apples dried at a microwave power of 100 W than in 200 W could be the result of gentler drying. It is likely that in such conditions the temperature of the material was lower, which reduced the risk of thermal degradation of phenolic compounds [51,74]. On the other hand, increasing the microwave power to 300 W was conducive to a significant reduction of the drying time (Figure 1), therefore the exposure of bioactive ingredients to negative conditions was relatively short, thanks to which an increased TPC retention was observed. This result is consistent with the literature data [51,57,75].





**Figure 5.** Response surface of total phenolic content (TPC) in apples treated with PEF prior to microwave-convective drying, depending on the microwave power (factor A) and specific energy intake (factor B).

The antioxidant activity, expressed as  $EC_{50}$  coefficient, of the apples untreated with PEF and dried at microwave power of 100, 200, and 300 W equalled 0.2775 ± 0.0064, 0.2005 ± 0.0245, 0.1829 ± 0.0273 mg d.m./mL, and 0.7519 ± 0.0205, 0.5550 ± 0.0388, 0.5981 ± 0.0482 mg d.m./mL for ABTS and DPPH assays, respectively. The reduced quadratic model allowed us to significantly predict (p < 0.05) the values of both parameters for apples treated with PEF and dried using the microwave-convective method. Moreover, a satisfactory fit of the model to the data obtained experimentally was observed, which was confirmed by the relatively high values of the determination coefficient ( $R^2 = 0.7966$  for ABTS assay,  $R^2 = 0.7608$  for DPPH assay) and the insignificant results of the lack of fit test (p > 0.05) presented in Table 3. ANOVA showed that both the  $EC_{50}$  ABTS and

 $EC_{50}$  DPPH of the tested samples significantly were influenced (p < 0.05) by microwave power (quadratic effect) and specific energy intake (linear effect). Based on Figure 6a,b, it can be seen that the values of  $EC_{50}$  ABTS and  $EC_{50}$  DPPH decreased with declining the amount of energy supplied during PEF treatment and with increasing the power of microwave from 180 to 300 W (between 100 and 180 W, a growth of these parameters was observed). Based on Equations (15) and (16), where the factors should be specified in actual values, it is possible to calculate the values of  $EC_{50}$  ABTS and  $EC_{50}$  DPPH of PEF-treated and microwave-convective dried apples. The highest value of TPC was observed in the sample, which was subjected to PEF treatment with a specific energy intake of 1 kJ/kg before microwave-convective drying in microwave power set at 300 W (Figure 5). As can be seen from Figure 6a,b, the above parameters also allowed to obtain the lowest  $EC_{50}$ ABTS and  $EC_{50}$  DPPH values, which indicates that this sample had the highest antioxidant activity. There are many reports of a relationship between antioxidant activity and total phenolic content [51,76-78]. Such conditions turned out to be the most optimal for the retention of bioactive ingredients in apples subjected to pulsed electric field treatment prior to microwave-convective drying.

 $EC_{50} ABTS = -0.132461 + 0.005379 \cdot Microwave Power + 0.061119 \cdot Specific Energy Intake$  $-0.000015 \cdot Microwave Power^2 - 0.004737 \cdot Specific Energy Intake^2$ (15)

 $EC_{50} DPPH = 0.018095 + 0.012041 \cdot Microwave Power + 0.063221 \cdot Specific Energy Intake$  $-0.000033 \cdot Microwave Power^2 + 0.000629 \cdot Specific Energy Intake^2$ (16)



**Figure 6.** Response surfaces of ability to scavenge: (a)  $ABTS^{\bullet+}$  radical cations (*EC*<sub>50</sub> ABTS); (b) DPPH<sup>•</sup> radicals (*EC*<sub>50</sub> DPPH) by apples treated with PEF prior to microwave-convective drying, depending on the microwave power (factor A) and specific energy intake (factor B).

## 3.4. Optimization of PEF Pretreatment and Microwave-Convective Drying

The most optimal process parameters, i.e., specific energy intake (pulsed electric field pretreatment) and microwave power (microwave-convective drying), were selected using RSM, based on specific criteria. Several responses were assigned a 'minimize' goal (drying time, water activity,  $H_{72h}$ ,  $EC_{50}$  ABTS, and  $EC_{50}$  DPPH). In the case of RR, SSL, and TPC, the main goal was to maximize these responses. Both factors and all responses had the same importance. The program identified the 13 most optimal combinations of process parameters under the above-mentioned criteria, and the best solution was selected based on

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the highest value of desirability. Specific energy intake equals 3.437 kJ/kg, and microwave power of 300 W turned out to be the most optimal parameters (desirability = 0.624). Such a combination of parameters would provide the following response values, predicted from the model: drying time:  $62 \pm 6$  min; water activity:  $0.188 \pm 0.010$ ;  $H_{72h}$ :  $1.2419 \pm 0.0055$ ; RR:  $2.2538 \pm 0.1158$ ; SSL:  $0.7275 \pm 0.0269$ ; TPC:  $1257.08 \pm 140.44$  mg GAE/100 g d.m.;  $EC_{50}$  ABTS:  $0.3045 \pm 0.0699$  mg d.m./mL;  $EC_{50}$  DPPH:  $0.9072 \pm 0.1666$  mg d.m./mL. Research by other authors [57] aimed at optimizing the microwave-convective drying of apples proved that the most desirable quality attributes of the obtained dried materials were achieved at the highest analysed microwave power.

## 4. Conclusions

Considering obtained results, hybrid drying is an interesting alternative to traditional drying methods. Undoubtedly, an important aspect, in addition to the appropriate selection of the drying method, is also the optimization and modeling of the process, thanks to which it is possible not only to reduce operating costs but also limit energy consumption and ensure obtaining a product with the most desirable quality.

The present research shows that it is possible to optimize the microwave-convective drying of apples with pulsed electric field pretreatment. PEF, by increasing the permeability of the cell membrane, led to a significant reduction in drying time (8–28%). No relationship was found between the amount of energy supplied during PEF pretreatment and the reduction of drying time; therefore, it is worth considering gentler conditions of treatment with an electric field to avoid overtreatment of the material. As it results from this study, the lowest amount of energy supplied during PEF processing (1 kJ/kg) allowed to obtain dried apples with the highest antioxidant activity (both  $EC_{50}$  ABTS and  $EC_{50}$  DPPH) and the highest total phenolic content among all PEF-treated and dried materials. Additionally, after drying these apples in the highest microwave power (300 W), the materials with the lowest total colour difference were obtained. This may be due to the partial inhibition of enzymes by PEF (e.g., polyphenol oxidase), which reduced the browning of the samples and limited the risk of oxidation of the bioactive ingredients. The amount of microwave energy supplied to the dried material can be increased to intensify the evaporation of water. At the same time, too high microwave power may cause partial burning of the dried samples. Increasing the microwave power from 100 to 200 W and from 200 to 300 W reduced the time of microwave-convective drying of apples by 45% in both instances. The use of the highest analysed microwave power (300 W) led to obtaining materials with the highest dry matter content, which means that the largest amount of water evaporated from them. Moreover, the highest microwave power, by significantly reducing the drying time and thus limiting the risk of thermal degradation of sensitive nutrients, allowed us to obtain dried apples with the highest antioxidant activity and TPC. Additionally, considering the water activity and the reconstitution properties of the dried apples (hygroscopicity and rehydration), the combination of the energy of 3.437 kJ/kg and microwave power of 300 W turned out to be the most optimal.

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