



PEF pre-treatment and ultrasound-assisted drying at different temperatures as a stabilizing method for the up-cycling of kiwifruit: Effect on drying kinetics and final quality

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ABSTRACT

The drying of food by-products, such as kiwifruit discards and surpluses, constitutes an interesting option as a means of stabilizing them for later use. This study assesses the combined influence of the drying temperature (40–70 °C), pulsed electric field (PEF) pretreatment (0–5000 V) and ultrasound (US; 0–50 W) application during the drying of kiwifruit. The increase in temperature, the use of PEF pretreatment or the US application during drying accelerated the process, but it was the combination of the three which led to the highest values of the effective diffusivity and the mass transfer coefficient. Over the range of different conditions studied, the functional and antioxidant properties of dried kiwifruit were not significantly affected, while the color presented some differences, mainly related to the L* and a* coordinates. Therefore, the combination of temperature, PEF and US allowed the processing time to be shortened without significantly affecting the final quality of kiwifruit. *Industrial relevance text:* Kiwifruit is one of the most globally popular fruits. Its high moisture content makes its by-products susceptible to chemical and microbiological degradation, so a drying step is needed to prolong its shelf-life and make up-cycling possible. To this end, hot air drying is one of the most widely-used techniques in the food industry, whose main drawback is a large amount of energy consumed. Therefore, there is a need to look for alternatives to reduce its economic and environmental impact and achieve a quality dried product. This study points out the combination of drying temperature, PEF pretreatments and ultrasound-assisted drying for the purposes of shortening the drying process while preserving the quality of the final product. These results, therefore, underline the promising nature of these techniques not only as regards their application in kiwifruit drying but also in the drying of other fruits and vegetables.

1. Introduction

The food industry generates large amounts of by-products, which represents a significant environmental and economic challenge. More and more importance is being given to the up-cycle of these by-products in order to favor circular economy systems (Klein, Nier, & Tamásy, 2022). Kiwifruit is a clear example of this. Millions of tons are produced annually throughout the world (Dai et al., 2022), and not all the production can be absorbed by the fresh market (discards and surpluses). This fruit has an excellent antioxidant profile and is a good source of

fiber (Kumar, Pipliya, & Srivastav, 2023), but its moisture content, exceeding 80% (Bhat et al., 2022), makes it very prone to suffering spoilage reactions.

In the food industry, convective drying is one of the most widespread dehydration techniques (Llavata, Picinelli, Simal, & Cárcel, 2022), with the main challenges being the lengthy process time and the large amount of energy consumed. Pretreatments using hyper or hypotonic solutions, either with or without ultrasound application, constitute one of the strategies whose aim is to partially modify the product structure and speed up the subsequent drying process. However, these options

Abbreviations: AA, ascorbic acid; AC, antioxidant capacity; BBD, Box-Behnken Design; D_{eff} , effective diffusivity (m^2/s); e_j , studentized residual; FRAP, ferric reducing ability power; GA, gallic acid; k, mass transfer coefficient ($kg\ w/m^2s$); MAE, Mean Absolute Error; MLR, multiple regression; ORC, oil retention capacity; PEF, pulsed electric field; RSM, Response Surface Methodology; R^2 , coefficient of determination; R^2_{adj} , adjusted coefficient of determination; SCE, specular component excluded; SW, swelling capacity; TPC, total phenolic content (TPC); US, ultrasound; VC, vitamin C; WRC, water retention capacity.

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generally entail large nutrient losses (Oliveira, Brandão, & Silva, 2016). In this sense, fast non-thermal techniques, such as pretreatments with pulsed electric fields (PEF), can overcome this drawback (Llavata, García-Pérez, Simal, & Cárcel, 2020). PEF technology consists of applying short-duration electrical pulses that can affect the structure of cell membranes, even generating temporary or permanent pores that could facilitate mass transfer. Thus, it has been reported that the PEF pretreatment led to drying time reductions of 27% in potatoes (Liu, Grimi, Lebovka, & Vorobiev, 2018), 30% in onions (Ostermeier, Giersemehl, Siemer, Töpfl, & Jäger, 2018), and even 57% in basil (Telfser & Gómez Galindo, 2019). On the other hand, the application of airborne ultrasound (US) during drying can also induce mechanical effects in the food matrix that can improve the drying rate without sample overheating (García-Pérez et al., 2023). In this sense, a significant shortening of the drying process has been reported in the ultrasound-assisted drying of food products (Huang et al., 2020).

PEF and US effects on food matrix structure could be complementary. In fact, these two technologies have been combined to improve processes such as extraction (Tzima, Brunton, Lyng, Frontuto, & Rai, 2021), frying (Ostermeier, Hill, Dingis, Töpfl, & Jäger, 2021) or microbial inactivation (Gomez-Gomez, Brito-de la Fuente, Gallegos, Garcia-Perez, & Benedito, 2021). In drying processes, some studies have reported the effectiveness of the application of combined pretreatments of PEF and US, this last applied in liquid media (Li et al., 2021; Rahaman et al., 2021; Rybak, Wiktor, Witrowa-Rajchert, Parniakov, & Nowacka, 2021). However, as Wang, Xiao, Ye, Wang, and Raghavan (2019) observed, in the case of kiwifruit, solid-liquid US assisted pretreatments lead to significant losses of soluble nutrients that can alter the final quality of the product.

To our knowledge, the combination of PEF pretreatment with airborne ultrasound-assisted drying has been only reported by Mello, Fontana, Mulet, Corrêa, and Cárcel (2021). These authors found a significant shortening of drying of orange peel, a porous product, when combining PEF pretreatments at two levels of intensity and an ultrasound-assisted drying process (50 W and 50 °C). On the other hand, it is widely described that when individually considered, the drying temperature, PEF pretreatment or US application, could affect the final

quality of the products (Neri et al., 2021; Szadzińska, Mierzwa, & Musielak, 2022). Therefore, it could be interesting to determine the combined influence of these factors.

Therefore, the goal of this study was to determine the combined effect of the drying temperature, PEF pretreatment and airborne ultrasound application during kiwifruit drying on the drying kinetics, as well as on the quality of the final product.

2. Materials and methods

2.1. Sample preparation

The kiwifruit (*Actinidia deliciosa*) used in these experiments was purchased in a local market in Valencia (Spain), with an initial moisture content of $82.4 \pm 0.9\%$. The samples were stored at 4 °C until the experiments were carried out.

2.2. Drying experiments

2.2.1. Pulsed Electric Field pretreatment (PEF)

Unpeeled kiwifruit pieces were pretreated individually in a parallelepiped chamber with two stainless electrodes (surface of 64 cm²) separated at a distance of 8.0 cm. The treatment chamber was filled up with tap water ($1035 \pm 28 \mu\text{S}/\text{cm}$ at 25 °C), covering the kiwifruit, and acting as an electric conductor between the electrodes (Fig. 1 A). A PEF generator (EPULSUS-PM1, Energy Pulse System, Lisbon, Portugal), operating in batch mode, provided monopolar near-rectangular shape pulses at a frequency of 10 Hz. Each treatment consisted of the application of 20 pulses of 25 μs each at a voltage of 0, 2500 and 5000 V, which corresponded to an energy input of 0, 13 and 50 J/kg.

2.2.2. Ultrasound-assisted drying

Drying experiments were carried out in a convective dryer equipped with an ultrasonic transducer (Fig. 1 B), previously described by Polachini et al. (2023). Thus, the kiwifruits, PEF pretreated or not, were peeled and cut into 6 mm slices with a sharp blade. For each drying run, 9 slices from the central part of the different fruits were placed in a

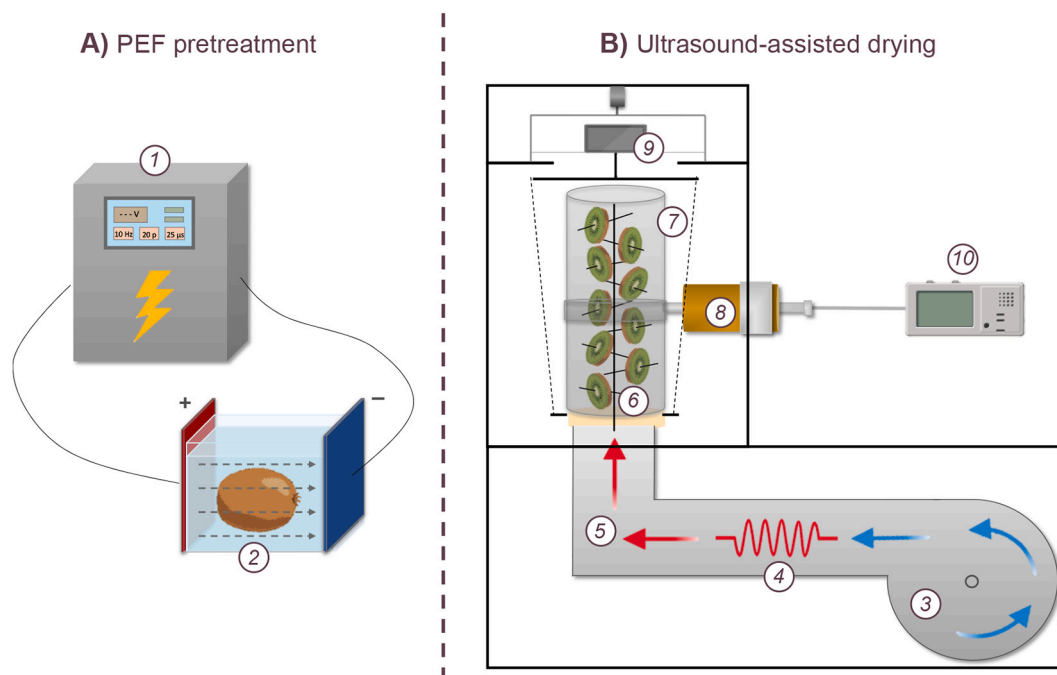


Fig. 1. Experimental set up. (A) Pulsed electric field treatment: 1) PEF generator, 2) electric current flow. (B) Ultrasound-assisted dryer: 3) fan, 4) thermal resistance, 5) hot air flow, 6) sample holder, 7) vibrating cylinder, 8) ultrasonic transducer, 9) automatic balance, 10) ultrasonic generator and impedance matching unit.

sample holder ensuring homogeneous air distribution. The drying experiments were carried out at an air velocity of 1 m/s and three different temperatures: 40, 55 and 70 °C. From an energy balance taking into account the air flow rate used, the energy needed to heat up and drive the air in each case was 239, 382 and 526 W. The weight of the samples was recorded automatically every 5 min. Airborne high intensity ultrasound (US) was applied through a cylindrical drying chamber which was excited with an ultrasonic transducer (21.8 kHz). Three levels of ultrasonic power applied were considered 0, 25 and 50 W. The drying process was stopped when no difference in the sample weight was recorded in 4 consecutive measurements.

After drying, the kiwifruit samples were stored in a dry environment with silica gel and protected from light until the analyses of the quality parameters.

2.2.3. Modeling of the drying kinetics

In order to quantify the influence of the temperature, PEF pretreatment and US application during the process, the drying kinetics were modeled using a model based on Fick's second law (Eq. (1)).

$$\frac{\partial W(x, t)}{\partial t} = D_{\text{eff}} \frac{\partial^2 W(x, t)}{\partial x^2} \quad (1)$$

where W was the moisture content (kg water (w)/kg dried matter (dm)), D_{eff} the effective diffusivity (m^2/s), t the drying time (s), and x the water transport direction (m). For this purpose, it was considered that the effective diffusivity (D_{eff}) was constant throughout the process, the initial moisture content was uniform in the sample (Eq. (2). Initial condition) and the solid was isotropic. Likewise, symmetric infinite slab behavior of the kiwifruit samples was assumed (Eq. (3), first boundary condition).

$$W(x, 0) = W_0 \quad (2)$$

$$\frac{\partial W(0, t)}{\partial x} = 0 \quad (3)$$

In addition, the external resistance to mass transfer was also considered. The mass transfer coefficient (k) was introduced in Eq. (1) as a second boundary condition (Eq. (4)).

$$-D_{\text{eff}} \rho_{\text{ss}} \frac{\partial W(L, t)}{\partial x} = k (a_w(L, t) - \varphi_{\text{air}}) \quad (4)$$

where ρ_{ss} was the dry solid density ($\text{kg dm}/\text{m}^3$), k the mass transfer coefficient ($\text{kg water}/\text{m}^2\text{s}$), a_w the water activity, L the half thickness of the layer, and φ_{air} the relative humidity of the air in each experiment. The equilibrium conditions were estimated from the desorption isotherm of the kiwifruit reported by Moraga, Martínez-Navarrete, and Chiralt (2006).

The model was solved through a finite difference method with Matlab software (The MathWorks, Inc., Natick, EE.UU.). The D_{eff} and k values were identified by minimizing the sum of the squared differences between the experimental and calculated moisture contents (SIMPLEX method). The goodness of fit of the model was quantified through the percentage of explained variance (% Var), described in Eq. (5).

$$\% \text{Var} = \left[1 - \frac{S_{xy}^2}{S_y^2} \right] \cdot 100 \quad (5)$$

where S_{xy}^2 is the standard deviation of the estimation, and S_y^2 is the standard deviation of the experimental data.

2.3. Quality parameters

2.3.1. Functional properties

Dried kiwifruit samples were ground and sieved (particle size <200 μm), obtaining a powder, in which different functional properties were

determined following the methodology described by Garau, Simal, Rosselló, and Femenia (2007). Thus, the swelling capacity (SW) was assessed by placing 0.20 ± 0.01 g of the powder in a test tube and evening it out at 10 mL with distilled water. After 24 h, the volume occupied by the powder was measured and compared with the initial volume. The difference was the SW, which was expressed as mL/g dm.

Moreover, 10 mL of still water or vegetable oil was added to 0.20 ± 0.01 g of kiwifruit powder in order to determine the water retention capacity (WRC) and oil retention capacity (ORC), respectively. After 24 h, it was centrifuged (Medifriger BL-S, P. Selecta, Spain) for 15 min at 10,000 rpm and 4 °C. Then the excess of liquid was removed and the solid weighed. The WRC and ORC were calculated as the difference between the initial and final weights, which was the amount of water, or oil, that had been retained in the kiwifruit powder. They were expressed as kg water or oil per kg of dry matter, respectively.

2.3.2. Antioxidant characteristics

The antioxidant characteristics of fresh and dried kiwifruit were measured in a previously obtained ethanolic extract. For this purpose, 1.000 ± 0.002 g of kiwifruit pieces was mixed with 20 mL of 96% ethanol. This was homogenized with an ultra-Turrax (T25 Digital, IKA, Germany) for 1 min at 13,500 rpm. After 24 h at 4 °C, the mixture was centrifuged (4000 rpm, 10 min) and filtered with microfiber glass paper (1.2 μm pore).

The total phenolic content (TPC) was determined using the Folin-Ciocalteu method, previously described by Gao, Bjork, Trajkovski, and Ugglá (2000). For this purpose, 100 μL of the previously prepared extract was mixed with 200 μL of Folin-Ciocalteu reagent (Sigma-Aldrich, Madrid, Spain) and 2 mL of distilled water. After 3 min of incubation at room temperature, 1 mL of 20% sodium carbonate (Sigma-Aldrich, Madrid, Spain) was added and incubated in darkness for one hour. Finally, the absorbance was measured in a spectrophotometer (Helios Gamma, Thermo Spectronic, Cambridge, UK) at 765 nm. The result was expressed as an equivalent concentration of gallic acid (GA).

The procedure described by Dani and Jagota (1982) was followed to assess the content of vitamin C (VC). Thus, 500 μL of the prepared extract were mixed with 500 μL of a $\text{C}_2\text{HCl}_3\text{O}_2$ solution (Panreac, Barcelona, Spain). Then, after 5 min at 4 °C, the mixture was filtered with a 0.45 μm pore filter. Finally, 2 mL of distilled water was mixed with 200 μL of the filtered mixture and 200 μL of a 1:10 solution of the Folin-Ciocalteu reagent. After 10 min of incubation, the absorbance was measured at 760 nm, identifying the mg of ascorbic acid (AA) per mg of dried matter.

The antioxidant capacity (AC) was determined using the Ferric Reducing Ability Power (FRAP) method (Benzie & Strain, 1996). Thus, the FRAP reagent was obtained by mixing 0.3 M acetate buffer (Panreac, Barcelona, Spain), 20 mM of $\text{FeCl}_3 \cdot 6\text{-H}_2\text{O}$ (Labkem, Barcelona, Spain) and 10 mM TPTZ (Sigma-Aldrich, Madrid, Spain) diluted in 40 mM HCl (Panreac, Barcelona, Spain), and incubating it for 30 min at 37 °C. Then, 900 μL of FRAP was mixed with 30 μL of the ethanolic extract and 30 μL of still water, all of which was incubated for 30 min at 37 °C until the spectrophotometer reading was taken at 595 nm. The results were obtained as mg Trolox/100 mg dm.

2.3.3. Color

The color of kiwifruit pulp before and after drying was measured through CIELAB coordinates with a colorimeter (CM – 2500d, Konica Minolta, Tokyo, Japan). The specular component excluded (SCE) was considered using a D65 illuminant reference and 10° opening angle. In this way, L^* (lightness/darkness), a^* (redness/greenness), b^* (yellowness/blueness) and the color difference (ΔE) between the fresh and dried samples were determined.

2.4. Experimental design and statistical analysis

The response variables (D_{eff} , k , SW, WRC, ORC, TPC, VC, AC, L^* , a^* ,

b^* and ΔE) as a function of drying temperature (T), pulsed electric field pretreatment (PEF) and ultrasound application (US) were studied using a rotatable second-order Box-Behnken Design (BBD). The experimental runs and nomenclature are shown in Table 1. The orthogonal least-squares calculation on factorial design data was computed using the following second-order polynomial expression (Eq. (6)).

$$Y = b_0 + \sum_{i=1}^n b_i X_i + \sum_{i < j=1}^n b_{ij} X_i X_j + \sum_{i=1}^n b_{ii} X_{ii}^2 \quad (6)$$

where Y was the different predicted response, n the number of independent variables, b_0 , b_i , b_{ij} and b_{ii} the coefficients of regressor of the mean, linear, interaction and quadratic terms, respectively, calculated by the least-square method and, X_i and X_j the independent variables. The statistical significance of the coefficients was verified by means of the F-test ($\alpha = 0.05$).

The adjusted coefficient of determination (R_{adj}^2) (Eq. (7)) and Mean Absolute Error (MAE) (Eq. (8)) were considered to evaluate the goodness of fit of the response surface.

$$R_{adj}^2 = 1 - \left(\frac{N-1}{N-M} \right) (1 - R^2) \quad (7)$$

$$MAE = \frac{\sum_{i=1}^N |X_{exp} - X_{cal}|}{N} \quad (8)$$

where N was the number of experimental data points, M the number of estimated model parameters, R^2 the coefficient of determination between the experimental and calculated values, X_{exp} and X_{cal} the experimental and calculated response variables.

Additionally, the Response Surface Methodology (RSM) was fitted using the function ‘‘Curve Fitting’’ of Matlab software.

2.4.1. Model validation

The studentized residual (e_j) was used to find and remove defective outliers. Any observations with e_j higher than a critical t-value ($|e_j| > |t_{N-1}^{(\alpha/2)}|$) were removed from the analysis.

The Shapiro-Wilk’s test and q-q plot were used to evaluate the normality of the residuals. Additionally, a multiple regression (MLR) was performed on square residuals to test variance homoscedasticity. All of the coefficient estimations and statistical assumptions were considered with a 95% confidence level and performed using Statgraphics Centurion XVIII (Manugistics, Inc., Rockville MD, USA). In this study, all of the residuals of the model regressions were adequately validated ($p > 0.05$); thus, the models of the variables were considered a valid tool for practical purposes.

Table 1
Box-Behnken experimental design and nomenclature.

Run	T (°C)	PEF (V)	US (W)	Nomenclature
1	40	2500	0	40 °C-2500 V-0 W
2	40	0	25	40 °C-0 V-25 W
3	40	5000	25	40 °C-5000 V-25 W
4	40	2500	50	40 °C-2500 V-50 W
5	55	0	0	55 °C-0 V-0 W
6	55	5000	0	55 °C-5000 V-0 W
7	55	2500	25	55 °C-2500 V-25 W
8	55	2500	25	55 °C-2500 V-25 W
9	55	2500	25	55 °C-2500 V-25 W
10	55	0	50	55 °C-0 V-50 W
11	55	5000	50	55 °C-5000 V-50 W
12	70	2500	0	70 °C-2500 V-0 W
13	70	0	25	70 °C-0 V-25 W
14	70	5000	25	70 °C-5000 V-25 W
15	70	2500	50	70 °C-2500 V-50 W

3. Results and discussion

3.1. Drying experiments

The experimental drying kinetics were determined in the conditions established with the rotatable second-order Box-Behnken Design (BBD) described in Table 1. Fig. 2 shows an example of the drying kinetics of each condition studied. As regards the factors considered, the air temperature had a clear effect on the drying rate of the kiwifruit samples. The increase in temperature led to a higher energy content in the medium, which improved not only the movement of the water molecules but also their change of state, from liquid to vapor, which speeded up the drying process. Thus, it took between 10 and 13 h for the experiments carried out at 40 °C to reach the end of the drying process; while at

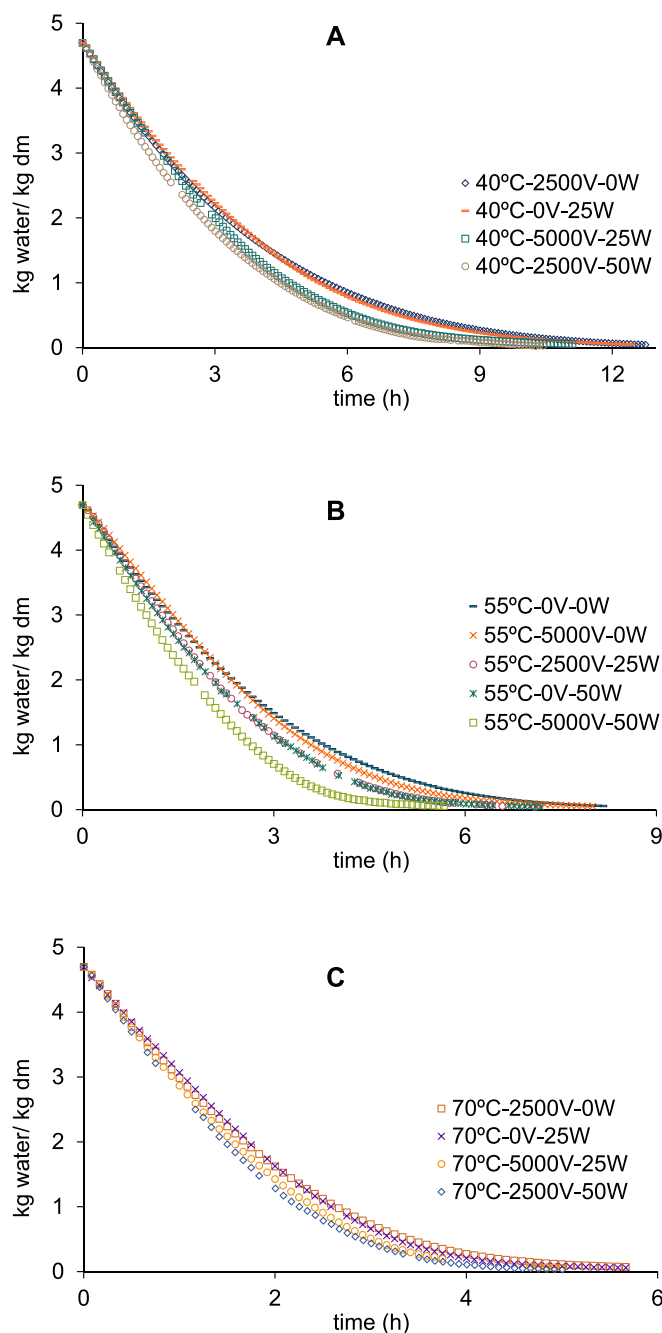


Fig. 2. Experimental drying kinetics of kiwifruit at the different conditions studied. Drying experiments carried out at 40 °C (A), 55 °C (B) and 70 °C (C).

70 °C, however, in no case did, the process take 6 h.

Another factor under study was the pretreatment with a pulsed electric field (PEF). This technique can cause structural changes due to the reorganization of the phospholipids of the cell membrane, giving rise to the formation of pores (Traffano-Schiffo et al., 2017). This electro- poration can facilitate the release of moisture from the intracellular space. In this study, some of the PEF pretreatments tested led to a slightly shortened drying process. Thus, taking the drying experiments performed at 55 °C as an example, the drying kinetics of PEF pretreated kiwifruit samples (55 °C-5000 V-0 W) were faster compared to the conventionally dried samples (55 °C-0 V-0 W), shortening the drying process by 5.3% (Fig. 2b). PEF has been shown to shorten the drying of different food products, such as carrots (Wiktor et al., 2016), apples (Matys, Dadan, Witrowa-Rajchert, Parniakov, & Wiktor, 2022) or basil (Thamkaew & Gómez Galindo, 2020).

The third factor considered, ultrasound (US) application during drying, speeded up the dehydration process. US can cause the formation of microcracks in the inner part of the samples and produces micro- agitation on the surface, which significantly facilitates mass transfer (Garcia-Perez et al., 2023). As an example, Fig. 2b shows that US- assisted drying at 55 °C (55 °C-0 V-50 W) shortened the drying time needed to reach a moisture content of 0.072 ± 0.001 kg water/kg dm by 19.1% compared to drying at 55 °C-0 V-0 W. In fact, the effectiveness of ultrasound-assistance during kiwifruit drying over a similar temperature range has recently been reported (Llavata, Femenia, Clemente, & Cárcel, 2023).

As can be observed in Fig. 2a and c (experiments carried out at 40 and 70 °C), the drying kinetics of samples which were pretreated with PEF (no US) and those which were subjected to ultrasound-assisted drying (no PEF) were superimposed. This fact may suggest that the drying enhancement induced by both technologies was similar. However, when both technologies were combined, there was an evident reduction in the drying time, significantly greater than when applied individually. The structural changes produced by the PEF pretreatment

could make the samples more prone to the US effects when applied during drying. This has been observed not only in the US-assisted convective drying of orange peel previously pretreated with PEF (Mello et al., 2021) but also when pretreatments with PEF and US application were combined before the convective drying of plum (Rahaman et al., 2021), carrot (Wiktor et al., 2019) or mushrooms (Li et al., 2021).

In addition, it should be noted that the increase in both the PEF voltage, from 2500 to 5000 V, and in the US power applied, from 25 to 50 W, produced an even greater shortening in the drying time of the samples dried at 55 °C. A higher intensity of PEF treatment can cause greater membrane disintegration (Huang et al., 2019) in the same way that more powerful US can increase its mechanical effects (Shi, Yang, Li, Wang, & Liu, 2020), being greater the synergy of these technologies. Thus, the drying time needed for the 55 °C-5000 V-50 W samples was in the same range as that of the conventional drying experiments per- formed at 70 °C.

3.2. Modeling

The effective diffusivity (D_{eff}) and mass transfer coefficient (k) were identified by fitting the proposed diffusive model to the experimental data (Table 2). The agreement between the calculated and experimental drying kinetics (Fig. 3) showed the goodness of fit. Moreover, the explained variance was >98.5% in every case.

The next step was the fitting of the polynomial models (Eq. (4)) to quantify the effect of the studied factors (temperature, PEF pretreatment and US application during drying) in both D_{eff} and k . The adequacy of these models was proven by the high values of R_{adj}^2 as well as the low values of MAE (Table 3).

The response surfaces obtained for D_{eff} underlined a significant ($p < 0.001$) influence of the drying temperature ($p < 0.001$); the higher the temperature, the greater the D_{eff} (Fig. 4a and b). Likewise, the applica- tion of the PEF pretreatment also had a highly significant ($p < 0.001$)

Table 2

Values (average \pm standard deviation) of the response variables (D_{eff} , k , SW, WRC, ORC, TPC, VC, AC, L^* , a^* , b^* , ΔE) after kiwifruit drying in different conditions: temperature, voltage of PEF pretreatments and power of US applied during drying.

Run	$D_{eff} \cdot 10^9$ (m ² /s)	$k \cdot 10^3$ (kg/ m ² s)	SW (mL/g dm)	WRC (kg w/kg dm)	ORC (kg oil/ kg dm)	TPC (%)	VC (%)	AC (%)	ΔE	L^*	a^*	b^*
40 °C-2500 V- 0 W	2.1 \pm 0.1	1.3 \pm 0.1	6.2 \pm 0.6	11.7 \pm 0.5	3.0 \pm 0.3	116 \pm 11	55 \pm 9	71 \pm 7	16 \pm 4	44 \pm 4	-2 \pm 2	49 \pm 7
40 °C-0 V-25 W	2.6 \pm 0.2	1.43 \pm 0.07	5.3 \pm 0.4	11.8 \pm 0.7	3.1 \pm 0.3	110 \pm 24	56 \pm 15	66 \pm 10	10.9 \pm 0.6	40 \pm 3	-2 \pm 2	57 \pm 2
40 °C-5000 V- 25 W	4 \pm 1	1.5 \pm 0.1	6.9 \pm 0.3	11 \pm 1	3.2 \pm 0.3	144 \pm 43	80 \pm 9	70 \pm 9	15 \pm 3	40 \pm 5	2 \pm 2	52 \pm 4
40 °C-2500 V- 50 W	3.7 \pm 0.7	1.69 \pm 0.06	7 \pm 1	10.4 \pm 0.4	3.1 \pm 0.3	138 \pm 8	66 \pm 8	61 \pm 5	12 \pm 2	41 \pm 5	0 \pm 3	49 \pm 2
55 °C-0 V-0 W	2.9 \pm 0.2	2.0 \pm 0.2	6 \pm 1	12.2 \pm 0.3	3.1 \pm 0.2	123 \pm 2	52 \pm 4	63 \pm 10	16 \pm 4	44 \pm 8	1 \pm 3	48 \pm 7
55 °C-5000 V- 0 W	6 \pm 1	1.9 \pm 0.1	7 \pm 1	11 \pm 1	3.2 \pm 0.2	115 \pm 4	57 \pm 13	63 \pm 5	17 \pm 2	45 \pm 3	3 \pm 2	48 \pm 5
55 °C-2500 V- 25 W	4.4 \pm 0.4	2.20 \pm 0.04	7 \pm 1	12 \pm 1	3.1 \pm 0.4	124 \pm 12	74 \pm 13	64 \pm 7	20 \pm 4	46 \pm 1	4 \pm 2	51 \pm 5
55 °C-2500 V- 25 W	4.4 \pm 0.4	2.12 \pm 0.02	7.1 \pm 0.1	11 \pm 1	3.0 \pm 0.5	145 \pm 16	69 \pm 7	77 \pm 5	18 \pm 3	45 \pm 1	4 \pm 2	50 \pm 10
55 °C-2500 V- 25 W	4.3 \pm 0.2	2.0 \pm 0.5	7 \pm 1	11 \pm 1	3.1 \pm 0.5	124 \pm 29	62 \pm 21	66 \pm 13	18.3 \pm 0.9	52 \pm 4	2.5 \pm 0.4	42 \pm 4
55 °C-0 V-50 W	4.2 \pm 0.9	2.5 \pm 0.2	7 \pm 1	12 \pm 1	3.1 \pm 0.3	141 \pm 15	74 \pm 9	66 \pm 9	17 \pm 1	38 \pm 7	3.3 \pm 0.6	48 \pm 5
55 °C-5000 V- 50 W	6 \pm 1	2.6 \pm 0.02	7.4 \pm 0.9	13 \pm 1	3.3 \pm 0.4	145 \pm 24	74 \pm 30	76 \pm 2	17 \pm 1	47 \pm 7	3.2 \pm 0.6	47 \pm 1
70 °C-2500 V- 0 W	6 \pm 1	2.7 \pm 0.2	5 \pm 1	12 \pm 0.2	3.1 \pm 0.2	95 \pm 15	67 \pm 23	64 \pm 18	19.8 \pm 0.9	49 \pm 2	4.3 \pm 0.4	56 \pm 9
70 °C-0 V-25 W	7 \pm 2	2.91 \pm 0.01	7 \pm 1	11 \pm 2	3.3 \pm 0.5	135 \pm 12	58 \pm 11	71 \pm 8	17 \pm 3	46 \pm 9	3 \pm 2	47 \pm 8
70 °C-5000 V- 25 W	7.2 \pm 0.9	3.16 \pm 0.03	6 \pm 1	12 \pm 2	2.7 \pm 0.2	142 \pm 15	66 \pm 2	78 \pm 11	19 \pm 3	50 \pm 3	5 \pm 1	52 \pm 9
70 °C-2500 V- 50 W	5 \pm 1	3.4 \pm 0.3	6.7 \pm 0.7	12 \pm 1	3.3 \pm 0.4	134 \pm 7	59 \pm 7	67 \pm 5	19 \pm 2	44 \pm 9	3.9 \pm 0.5	46 \pm 0.8

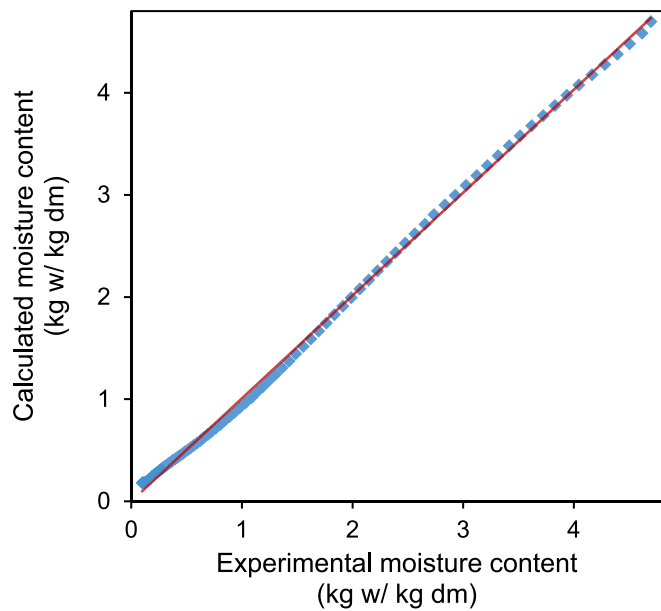


Fig. 3. Experimental vs calculated moisture content of kiwifruit, dried at 55 °C, with a PEF pretreatment of 2500 V and with application of ultrasound during drying at 25 W (experiment 55 °C-2500 V-25 W).

effect on D_{eff} . The quadratic effect of PEF was positive (Table 3), so from a certain point the curvature of the RSM was upward, as the D_{eff} values were higher. Thus, as can be observed in Fig. 4a, only when a voltage threshold was overcome was a significant increase in D_{eff} identified at 40 °C. In this sense, when drying apples pretreated with PEF, Wiktor et al. (2013) observed that the experiments carried out at low electric field strength and using a small number of pulses (5 kV/cm and 10 pulses) exerted no significant influence on the D_{eff} values. On the contrary, the more intense PEF pretreatments (10 kV/cm and 50 pulses) induced a significant increase in the D_{eff} . In these conditions, the PEF pretreatment could create not only reversible short-lived pores, but irreversible pores that promoted mass transfer. Thus, several authors have reported an increase in the D_{eff} in drying experiments carried out with PEF pretreated samples (Mirzaei-Baktash, Hamdami, Torabi, Fallah-Joshaqani, & Dalvi-Isfahan, 2022; Rahaman et al., 2019; Wiktor et al., 2021).

Table 3

Second order polynomial results for describing the response variables as a function of independent variables and statistical results.

Coefficients	D_{eff}	k	SW	WRC	ORC	TPC	VC	AC	ΔE	L^*	a^*	b^*
b_0	-3.78	-0.65	6.06	2.30	13.30	113	11.27	78.87	-14.94	36.01	-28.74	91.25
b_1 (T)	0.13***	0.05***	ns	ns	-0.02 ^{ns}	ns	ns	ns	0.95***	0.23**	0.84***	ns
b_2 (PEF)	-6.43 × 10 ^{-5***}	ns	ns	ns	-2.49 × 10 ^{-4ns}	ns	ns	ns	1.68 × 10 ^{-3ns}	-7.60 × 10 ^{-4ns}	1.13 × 10 ^{-3*}	ns
b_3 (US)	0.09*	4.05 × 10 ^{-4***}	0.02*	ns	-0.11 ^{ns}	0.59***	ns	ns	ns	-0.18 ^{ns}	0.21*	ns
b_{12}	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
b_{13}	-1.39 × 10 ^{-3*}	ns	ns	ns	1.61 × 10 ^{-3*}	ns	ns	ns	ns	ns	-2.35 × 10 ^{-3*}	ns
b_{23}	ns	ns	ns	ns	9.45 × 10 ^{-6*}	ns	ns	ns	ns	-4.97 × 10 ^{-5*}	ns	ns
b_1^2	ns	ns	ns	ns	ns	ns	ns	ns	-7.14 × 10 ^{-3*}	ns	-5.56 × 10 ^{-3*}	ns
b_2^2	8.97*	ns	ns	ns	ns	ns	ns	ns	-2.61 × 10 ^{-7*}	ns	ns	ns
b_3^2	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns	ns
R_{adj}^2 (%)	77.15	95.60	9.50	0	13.92	29.66	3.8	0	43.53	28.76	66.74	0
MAE ¹	0.56	0.11	0.83	0.24	0.78	12.41	8.69	6.70	1.86	3.48	1.03	4.54
Lack of fit (p-value)	0.62 ^{ns}	0.78 ^{ns}	0.77 ^{ns}	0.36 ^{ns}	0.41 ^{ns}	0.68 ^{ns}	0.09 ^{ns}	0.38 ^{ns}	0.54 ^{ns}	0.67 ^{ns}	0.80 ^{ns}	0.42 ^{ns}

¹ Mean absolute error (MAE) in units of each response variable. Uppercase characters indicate statistical significance. ns: non-significant. * $p < 0.05$. ** $p < 0.01$. *** $p < 0.001$.

US application during drying was also a significant factor ($p < 0.05$) in the D_{eff} value (Fig. 4b and c). At an internal level, an improvement in mass transfer caused by US was likely due to effects, such as the sponge effect and microcracks formation, brought about by the induced mechanical stress. In addition, the interaction between the drying temperature and US was negatively significant ($p < 0.05$), which meant that the lower the temperature, the more effective US application was at increasing D_{eff} (Dadan, Nowacka, Wiktor, Sobczynska, & Witrowa-Rajchert, 2020).

As for the mass transfer coefficient (k), it was significantly ($p < 0.001$) affected by the temperature (Table 3). A high temperature can enhance the evaporation process on the kiwifruit surface and its transport to the surroundings. US also significantly ($p < 0.001$) increased the mass transfer coefficient (Table 3). This can be attributed to a decrease in the boundary layer of diffusion due to the generation of microstirring at interfaces (Garcia-Perez et al., 2023), reducing the external resistance to mass transfer. In contrast, the application of PEF pretreatments was not statistically significant. The electroporation that took place in the cells of the external layer of the samples was not intense enough to induce a significant effect on the external mass transfer resistance.

3.3. Quality parameters

3.3.1. Functional properties

Drying can affect the bioactive compounds present in the kiwifruit and, thus, their functional properties. Therefore, the swelling capacity (SW) of the dried kiwifruit ranged from 5.3 to 7.4 mL/g dm, depending on the conditions studied (Table 2). After the fit of the polynomial model (Eq. (4)), it was found that the only factor that significantly affected ($p < 0.05$) the SW was the US application during drying (Table 3). Thus, it was observed that the increase in ultrasonic power led to a rise in the SW values. The mechanical effects of US can cause cell rupture and bring about the opening of the structures, which leads to an increase in the electrical charge and a greater exposure of the hydrophilic groups (Khanpit, Tajane, & Mandavgane, 2022). Furthermore, the use of high temperatures can promote cell damage in certain polysaccharides, while low temperatures usually lead to longer drying times that can cause structural shrinkage (Borsini, Llavata, Umaña, & Cárcel, 2021). Although the effect of the temperature was not significant, the response surfaces (Fig. 5a and b) showed that the highest SW values were obtained at intermediate temperatures. These results agree with those reported by Llavata et al. (2022) for apple pomace.

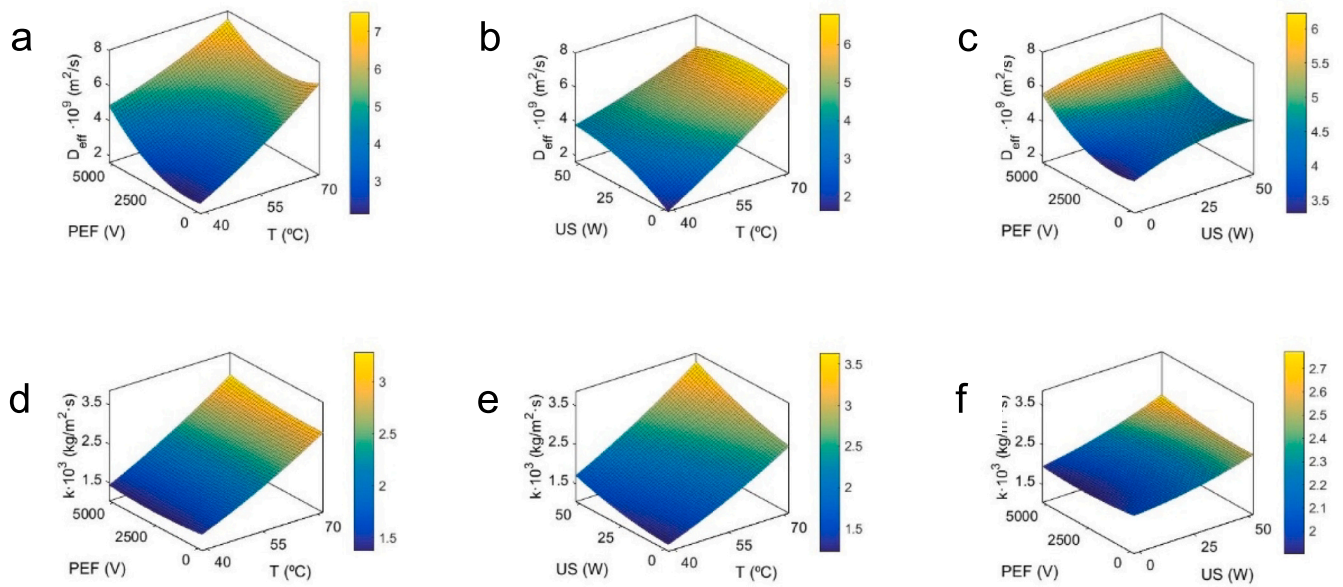


Fig. 4. Response surface plots for effective diffusivity (D_{eff}) and mass transfer coefficient (k) identified during drying of kiwifruit. Process variables considered: Drying temperature (T), voltage applied in PEF pretreatment (PEF) and power of ultrasound applied during drying (US).

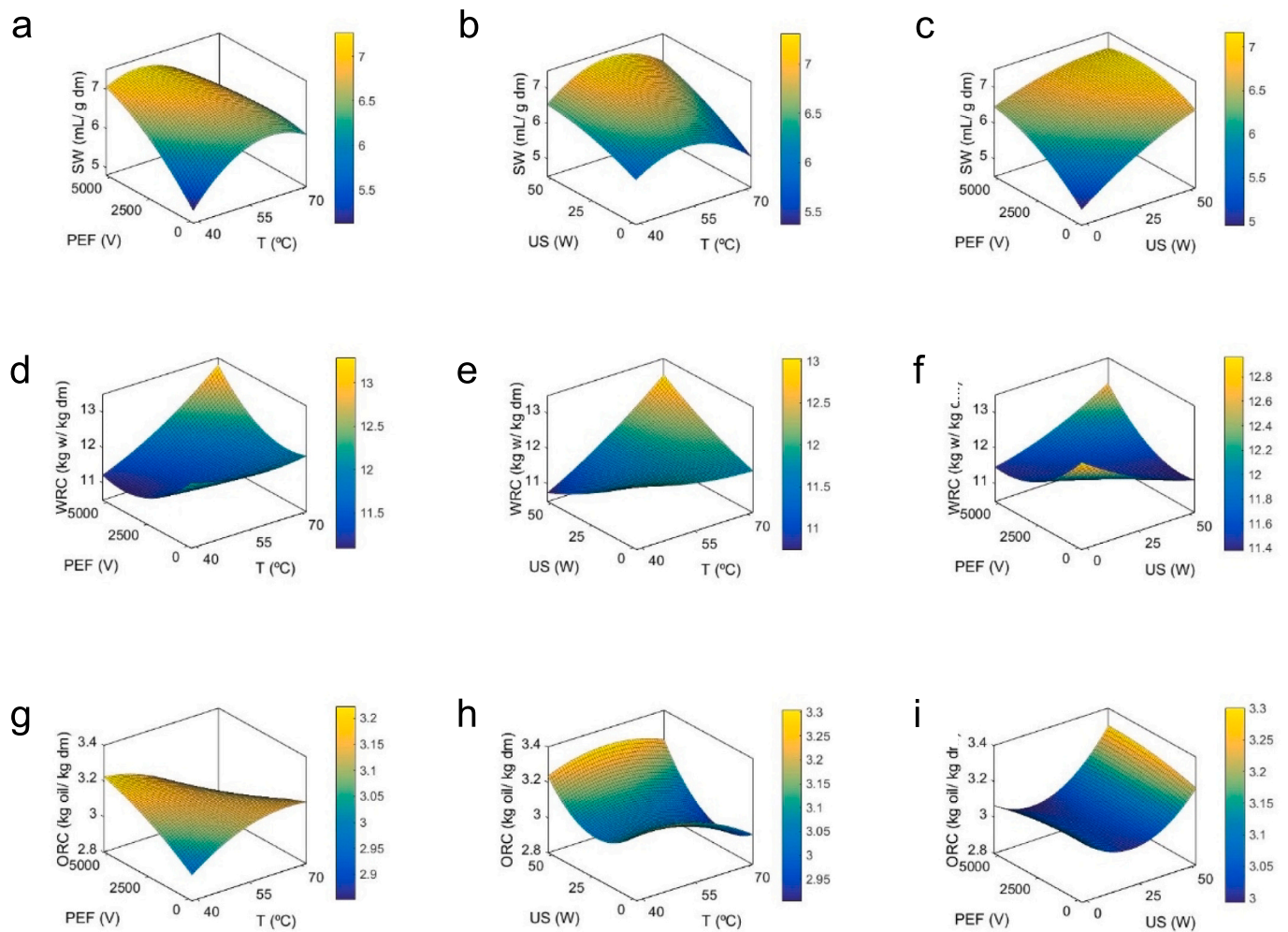


Fig. 5. Response surface plots for functional properties of kiwifruit after drying: swelling capacity (SW), water retention capacity (WRC) and oil retention capacity (ORC). Process variables considered: drying temperature (T), voltage applied in PEF pretreatment (PEF) and power ultrasound applied during drying (US).

As far as the water retention capacity (WRC) of dried samples is concerned, the measured values were between 10.4 and 13 kg water/kg dm (Table 2). The process conditions tested were found to have no clear influence on this property and none of the factors studied were significant (Table 3).

The oil retention capacity (ORC) ranged from 2.7 to 3.3 kg oil/kg dm (Table 2). In this case, the statistical analysis (Table 3) indicated that the interaction between temperature and US was significant ($p < 0.05$). This is reflected in Fig. 5h, in which a maximum ORC value was identified at the highest level of US applied and at an intermediate temperature of those tested. In the same way that US can increase the exposure of hydrophilic groups, as mentioned previously, they also expose hydrophobic groups, increasing their union with the oil. Thus, Vallespir, Crescenzo, Rodríguez, Marra, and Simal (2019) found an increase in ORC when US was applied during the drying of mushrooms. In addition, the interaction between the PEF and US was also statistically significant ($p < 0.05$), as shown in Table 3 and Fig. 5i. The structural changes produced by PEF could cause a branching of the polymers that constitute the kiwifruit fiber. Wang et al. (2023) attributed the increase in the ORC of PEF-treated peanut shell to an rise in the porosity of the structure leading to a more complex surface area facilitating oil binding. PEF could also cause an unfolding of the proteins present in the kiwifruit seeds that increased the interaction between the molecules (Bran et al., 2023). This could favor the subsequent action of US when applied during drying.

However, as indicated by R_{adj}^2 and MAE (Table 2), the significance of the fit of the polynomial model within the functional properties considered was limited. It should be noted that kiwifruit is a highly

variable, very heterogeneous product, as can be observed in the high values of standard deviation found (Table 2). This can mask the effects of the combination of temperature, PEF pretreatments and US application during drying.

3.3.2. Antioxidants

Table 2 shows the retention percentages of TPC, VC and AC of kiwifruit dried in the different drying conditions. The fresh kiwifruit samples presented an initial phenolic compound content (TPC) of 5.3 ± 0.9 mg GA/100 g dm, which increased after drying. The high temperatures used in the drying process can affect the structure of the cells, promoting the release of phenolic compounds (Özcan et al., 2020). Moreover, some authors have attributed this increase in TPC to a response of the biological material to the stress induced by the loss of moisture during drying, which can generate new phenolic compounds as a defence mechanism (Roshanak, Rahimmalek, & Goli, 2016).

US application during drying was the only factor that significantly affected the TPC (Table 3). The increase in US power led to a greater retention of the phenolic compounds. These results agree with those reported previously, where it is shown that ultrasound-assisted drying could improve the TPC retention percentages in kiwifruit (Liu, Zeng, Guo, & Sun, 2019) or mushrooms (Szadzińska et al., 2022). US can promote the formation of new phenolic compounds through the combination with other compounds and also activate secondary metabolic pathways. Moreover, US could also contribute to the partial inactivation of oxidative enzymes, thus preventing their degradative action on polyphenols (Vallespir, Rodríguez, Cárcel, Rosselló, & Simal, 2019).

As for vitamin C (VC), the initial content of kiwifruit samples was 1.5

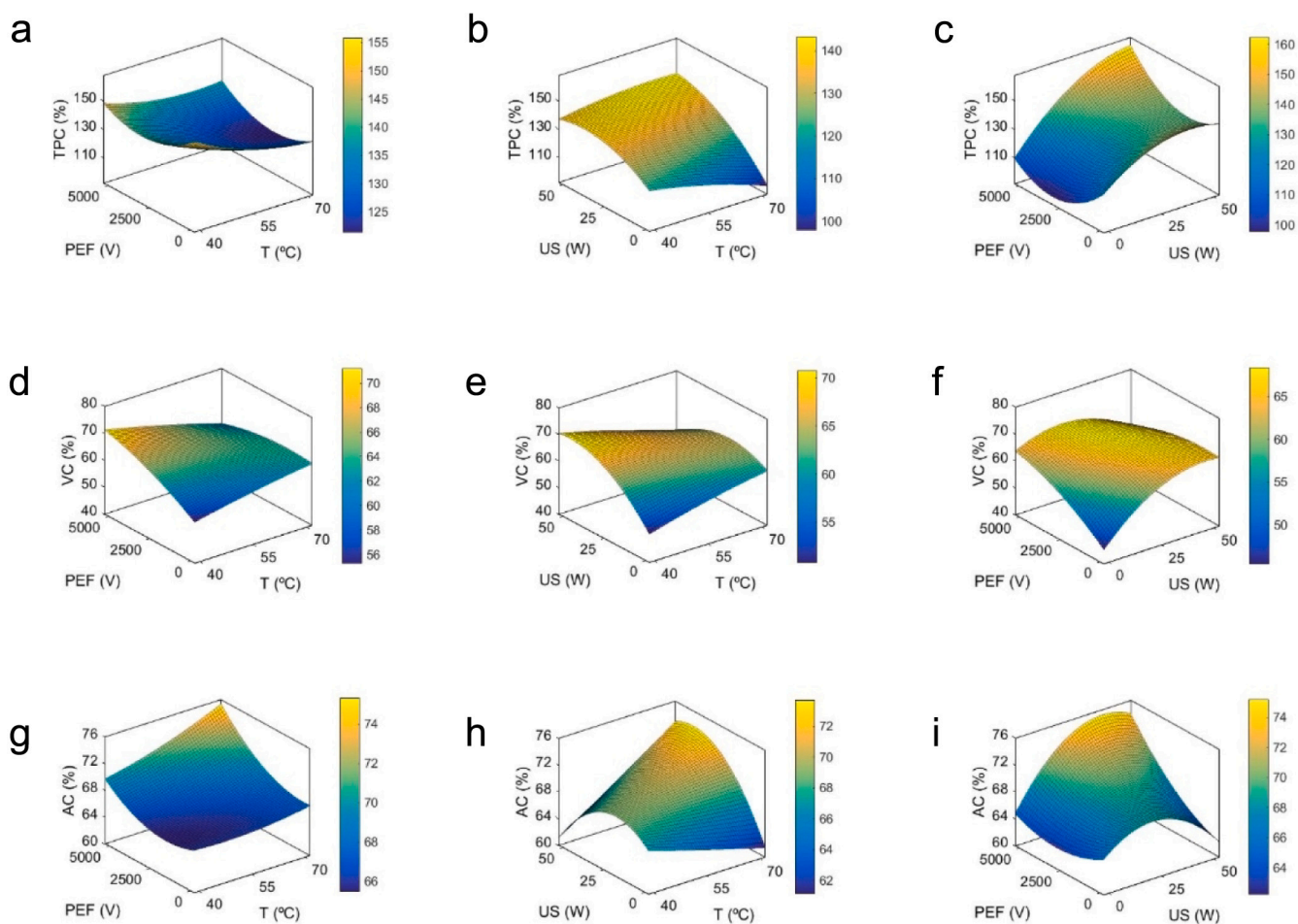


Fig. 6. Response surface plots for antioxidant characteristics of dried kiwifruit: total phenolic content (TPC), vitamin C (VC) and antioxidant capacity (AC). Process variables considered: drying temperature (T), voltage applied in PEF pretreatment (PEF) and power ultrasound applied during drying (US).

± 0.4 mg AA/g dm. VC was the antioxidant property that most decreased after drying due to its high degree of sensitivity to both temperatures and oxygen exposure during drying. The final VC content of the conventionally-dried samples (no PEF, no US) was greater at the highest temperature tested, 70 °C (Fig. 6d and e). Thus, the use of high temperatures could degrade VC, but, at the same time, shorten the drying time. Moreover, the PEF pretreatment reduced the drying time but could also enhance both the degradation of the ascorbic acid and the improvement of the ascorbic oxidase activity (Wiktor, Dadan, Nowacka, Rybak, & Witrowa-Rajchert, 2019). US application could also accelerate the drying, but its mechanical effects could cause greater cell damage (Cárcel, Castillo, Simal, & Mulet, 2019). This set of opposing effects meant that the VC after drying cannot be explained by a single factor. In this sense, no variables have been found to have a significant effect on the VC retention (Table 3). Additionally, the high values of standard deviation (Table 2) indicated a great experimental variability that can mask any effect of the factors under study. Previously, other authors did not find any differences in VC content when drying PEF pretreated spinach (Yamakage et al., 2021) or blueberries (Yu, Jin, & Xiao, 2017), or when PEF pretreatments were combined with the ultrasound-assisted drying of orange peel (Mello et al., 2021).

The antioxidant capacity (AC) of the fresh kiwifruit samples was 37.1 ± 6.1 mg Trolox/100 g dm. As happened in the case of VC, AC also decreased after drying. AC is a measurement that encompasses the synergistic and antagonistic effects of the different antioxidant compounds present in the samples (Llavata et al., 2022), such as phenolic compounds and ascorbic acid. Similarly to VC, the studied variables were observed to have no clear influence on the AC retention. This may also be seen in the literature, in which contradictory results are found (Lammerskitten et al., 2020; Mirzaei-Baktash et al., 2022; Neri et al., 2021). Thus, Rybak et al. (2022) reported that both PEF and US can induce the formation of free radicals and reactive oxygen species, which, combined with the enhancement of their penetration into broken cells, could lead to a decrease in AC. On the contrary, these technologies not only reduce oxygen exposure time, but could also produce a partial inhibition of oxidative enzymes (Wiktor et al., 2021).

In general, it could be said that the behavior and the activity of antioxidant compounds during drying is complex and cannot easily be described by the variables studied, much less if the high degree of natural variability observed in kiwifruit is also taken into account. Thus, the mathematical models obtained to describe them exhibited limited feasibility (Table 3).

3.3.3. Color

In terms of the quality of a dried product, color is one of the most important parameters and exerts a great influence on consumer acceptance. The fresh kiwifruit samples presented L^* , a^* and b^* values of 37 ± 5 , -9 ± 2 and 45 ± 8 , respectively. Drying induced significant changes in the kiwifruit color, as clearly shown by the ΔE values (Table 2). These differences in color were mainly attributed to changes in the coordinates of L^* and a^* , since the values of b^* remained practically unchanged. In fact, no significant influence of the studied variables was found for this parameter (Table 3).

On the contrary, the luminosity (L^*) of the kiwifruit samples increased after drying and was significantly ($p < 0.01$) affected by the drying temperature (Table 3). During the drying process, the pigments present in kiwifruits can be thermally degraded, producing the discoloration of the fruit (Taghinezhad, Kaveh, & Szumny, 2021). Thus, it was observed that the higher the temperature, the greater the L^* value. The a^* coordinate also increased after drying, so that the fresh green samples became brownish. This may be particularly related to the degradation of chlorophyll, which is a very heat-sensitive compound (Telfser & Gómez Galindo, 2019). In fact, the drying temperature significantly affected a^* at lineal ($p < 0.001$) and quadratic ($p < 0.05$) levels (Table 3). In addition, the PEF pretreatment and ultrasound-assisted drying also

influenced the a^* value. The electroporation caused by PEF and the cell disruption caused by US could promote the release of certain kiwifruit pigments and oxidative enzymes that favored the browning reactions (Nowak & Jakubczyk, 2022; Tylewicz et al., 2020). In this sense, some authors have reported an increase in the a^* coordinate after the drying of PEF pretreated onion (Ostermeier, Parniakov, Töpfl, & Jäger, 2020) or parsnip (Alam, Lyng, Frontuto, Marra, & Cinquanta, 2018). It should also be noted that the interaction between temperature and US was significantly ($p < 0.05$) negative. Thus, the US applied at the lowest temperatures tested led to a greater increase in a^* than when applied at 70 °C (Fig. 7h). This could be attributed to the fact that at 40 °C, the enzymes that could be released due to the cell rupture caused by US would not be fully inactivated, while at 70 °C they would.

4. Conclusion

The combination of temperatures, PEF pretreatments (voltage) and ultrasound (power applied) during kiwifruit drying was assessed. Individually considered, the three factors had a clear impact on drying kinetics, but the combination of both PEF and US technologies provided the shortest drying. From the modeling, it was observed that the rise in the drying temperature and US power applied increased both the D_{eff} and k . The influence of the PEF pretreatments mainly focused on the rise in D_{eff} , reducing the internal resistance to mass transfer. The polynomial models showed that the three factors considered, temperature, PEF pretreatment and US application, significantly affected both drying kinetics parameters, D_{eff} and k .

The quality of the final product was assessed through the functional, antioxidant and color properties. The PEF pretreatments and US application particularly enhanced the oil retention capacity. In the same way, US increased the retention of TPC. The color of the dried samples was mainly affected by changes in the L^* and a^* coordinates. The variable that most affected the color was temperature, although PEF and US also promoted a greater browning of the dried kiwifruit. However, on the whole, the polynomial model provided a poor fit to the quality parameters. The high degree of experimental variability found for some of them and the possible opposite effects induced by the three factors studied could mask their effects.

Overall, it could be concluded that the combination of the PEF pretreatment and ultrasound-assisted drying significantly shortened the drying process without inducing significant changes in the final quality of the product.

Author statement

Manuscript “PEF pre-treatment and ultrasound-assisted drying at different temperatures as a stabilizing method for the up-cycling of kiwifruit: effect on drying kinetics and final quality”.

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CRediT authorship contribution statement

B. Llavata: Writing – original draft, Validation, Software, Methodology, Investigation, Formal analysis, Data curation. **G.A. Collazos-Escobar:** Writing – original draft, Validation, Software, Investigation, Formal analysis, Data curation. **J.V. García-Pérez:** Writing – review &

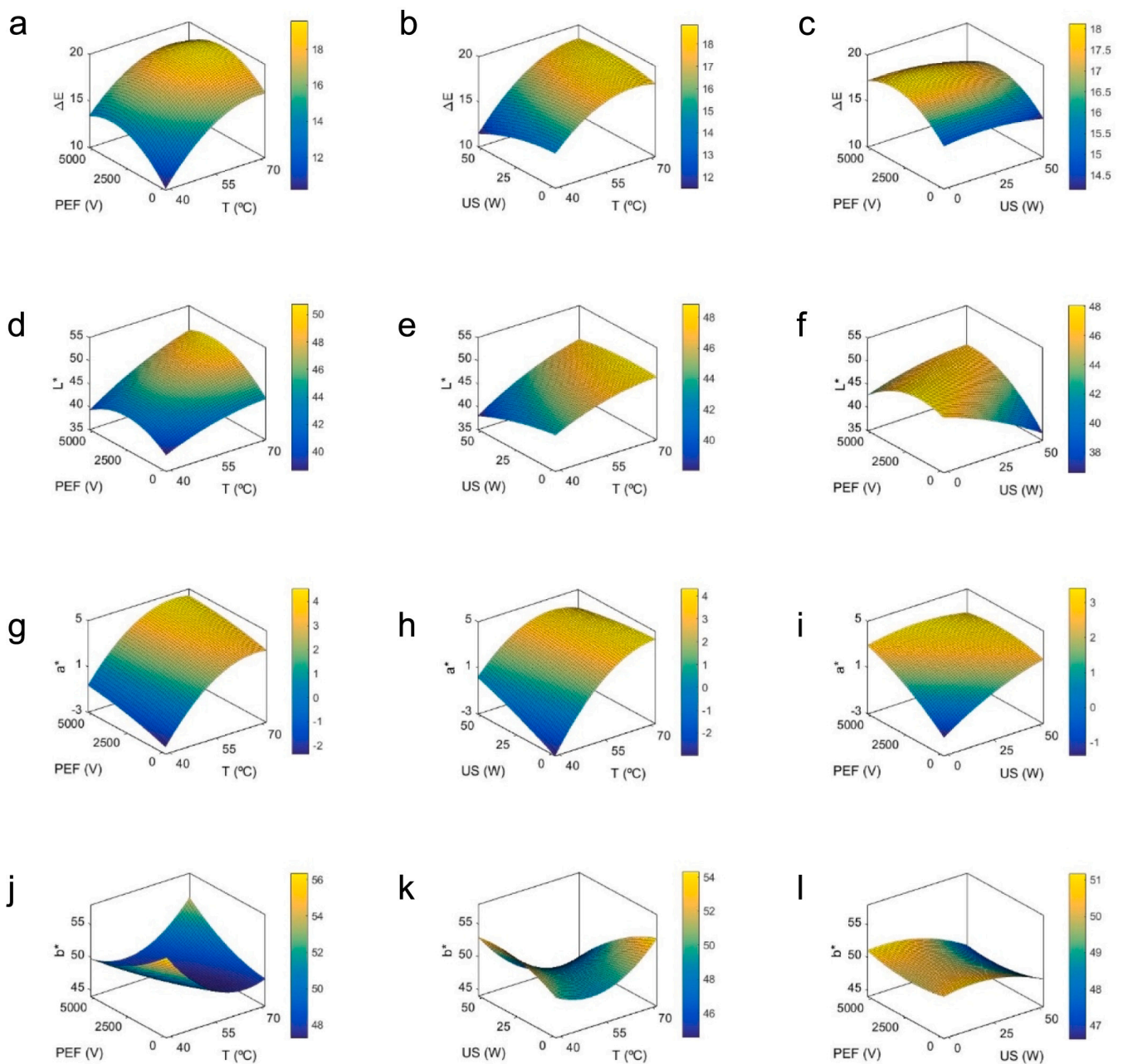


Fig. 7. Response surface plots of the coordinates L^* , a^* and b^* and the color difference (ΔE) between the fresh and dried kiwifruit samples. Process variables considered: Drying temperature (T), PEF pretreatment (PEF) and ultrasound application during drying (US).

editing, Supervision, Formal analysis, Data curation, Conceptualization. J.A. Cárcel: Conceptualization, Methodology, Validation, Formal analysis, Resources, Writing – review & editing, Methodology, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

None.

Data availability

Data will be made available on request.

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