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Non-Thermal Pretreatments for Hot-Air Drying of Whole Black Chokeberry (*Aronia melanocarpa*): Comparison of Cold Plasma and Indirect Ultrasound

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Abstract

Whole black chokeberries, with their surface wax and firm structure, dry slowly. Atmospheric-pressure cold plasma for 150 or 300 s (CP2.5 and CP5) and indirect ultrasound for 30 min (IUS) were compared before hot-air drying of whole fruit at 50, 60 and 70 °C, with drying duration fixed within each temperature. CP2.5 gave the strongest kinetic response, reducing the time to a moisture ratio of 0.10 (t_{10}) by 19–25%, against 12–17% for CP5 and 11–13% for IUS, and increasing the apparent effective moisture diffusivity by 24–34%. It also gave the lowest mean CIELAB browning index, 64–72% below the control, and the highest mean phenolic, flavonoid and anthocyanin contents among the hot-air-dried groups, with total flavonoid content significantly higher than the control at all temperatures; all remained below the freeze-dried reference. CP5 gave the lowest and IUS the highest shrinkage, and IUS did not differ from the control in composition or antioxidant capacity. Raising the temperature from 50 to 70 °C reduced control t_{10} by 59%; anthocyanin means were 8–15% lower at 70 °C than at 50 °C. The Arrhenius slopes of the four groups did not differ significantly. Extending plasma exposure reduced the kinetic benefit; the compositional advantage of CP2.5 over CP5 was not statistically demonstrated.

Keywords: activation energy; anthocyanins; cold plasma; drying kinetics; effective moisture diffusivity; indirect ultrasound; thin-layer modelling; whole black chokeberry (*Aronia melanocarpa*)

1. Introduction

Black chokeberry [*Aronia melanocarpa* (Michx.) Elliott, Rosaceae] fruits are rich in anthocyanins, proanthocyanidins and hydroxycinnamic acids [1]. Their sourness, bitterness and astringency limit fresh-fruit acceptance [2], and the berries are mainly processed into juice, powder and dried fruit [3]. Although hot-air drying is simple to operate [4], whole chokeberries require long drying times [4,5]. Surface wax [6] and firm fruit structure [5] are considered the major resistances to moisture transfer, although their individual contributions have not been measured. Drying whole fruit to 0.05 kg water/kg dry matter required 23 h at 50 °C and 11 h at 70 °C, with greater polyphenol loss at the lower temperature [7]. In another study, 24 h of convective drying at 65 °C gave a slightly lower total phenolic content than fresh fruit (77.7 versus 79.7 mg gallic acid equivalents g^{-1} dry weight), while freeze-dried fruit did not differ from fresh fruit [8]. Pretreatments intended to lower this resistance without a deliberate heating step are therefore of interest.

Pretreatments can increase heat and mass transfer, shorten drying and limit quality losses [9]. Alkaline-emulsion and hot-water dipping alter the surface wax [6], but introduce

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a chemical and a thermal processing step, respectively. Ultrasound can facilitate moisture migration through cavitation and sponge effects that may create tissue microchannels [9]. Cold plasma can modify fruit surfaces through reactive species; wax erosion, a reduced water contact angle and micropores or microcracks have been reported in grapes and other plant materials [10] and in apple slices [11]. Nevertheless, a systematic review noted that direct evidence linking these surface changes to the drying response was lacking [10], and no corresponding surface measurements have been reported for whole chokeberry treated with the plasma-jet system used here. Plasma reactive species may also oxidise phenolic compounds [12]. Treatment conditions therefore need to balance improved moisture transfer against quality changes, and the response depends on the equipment, the treatment parameters and the food matrix [9,13].

In chokeberry, potassium carbonate–olive oil and hot-water dipping generally reduced the final moisture content and water activity of the dried fruit [6]. Ultrasound has been applied to whole berries immersed in isotonic mannitol solution at 25 °C before hot-air drying [5], and to berries held in a beaker placed in a bath at 50 °C before vacuum drying [4]. In a further study, berries were sonicated in an ultrasonic bath for 1–20 min and subsequently dried as halves under infrared; the shortest drying times were obtained after 10 min of pretreatment, and longer treatments gave no further reduction [14]. These studies do not describe the response of whole chokeberry to ambient-temperature indirect sonication in water before hot-air drying. Previous chokeberry studies selected the modified Page [2], Henderson–Pabis [15], Logarithmic [4] and Midilli–Küçük [16] thin-layer models under different drying conditions. A lower Arrhenius activation energy after ultrasound pretreatment [5] and effective moisture diffusivities for hot-air-dried whole fruit [4,5] have also been reported, and these studies serve as reference points for the kinetic and quality variables evaluated here.

Gliding arc plasma shortened hot-air drying of whole grapes at 60 °C by up to 26.27% [17]. In jujube slices, the shortest plasma exposure gave the highest effective diffusivity among the treated groups, whereas the longest produced values below the control at all three drying temperatures [18]. Conversely, whole-blueberry diffusivity increased with exposure time and power over the ranges tested [19]. Radio-frequency plasma shortened whole-blueberry drying by 50%, compared with 8–17% after blanching, freeze–thaw, alkaline dipping or ultrasound in sodium carbonate solution [20]. Reported plasma exposures range from tens of seconds [17] to several minutes [21]. In chokeberry, plasma has been used for whole-fruit decontamination [22] and for extraction from dried powder with the plasma-jet model used here [23]. Plasma pretreatment has been examined at three drying temperatures for sliced jujube [18], sliced jackfruit [24] and shiitake mushroom [25]. The cited whole-berry studies used one drying temperature [17,20], so they do not describe how the plasma response changes with drying temperature. Comparisons of ultrasound and plasma before hot-air drying at 60 °C have also involved sliced products. In yam slices sonicated in a water-filled beaker, the relative performance of the two pretreatments depended on the ultrasound power [26]. In apple slices, 30 min of bath sonication and 60 s of low-pressure plasma shortened drying by 46.7% and 33.3%, respectively, with smaller or no reductions at longer plasma exposures, and both treatments reduced the surface water contact angle [11]. Neither study examined a whole, wax-covered fruit, so the relative response of such a fruit to the two pretreatments cannot be inferred from them.

Drying each group to a common final-moisture criterion results in different drying durations [20], so quality differences are difficult to separate from the unequal thermal exposure. Here, drying duration was fixed within each temperature to compare moisture removal and the resulting product quality under the same drying-air temperature and residence time. Final moisture content was therefore allowed to differ, and the quality

comparisons apply to products obtained under the same drying programme rather than at equivalent final moisture contents.

This study compared atmospheric-pressure cold plasma (150 and 300 s) and indirect ultrasound (fruit immersed in water in a beaker placed in an ultrasonic bath for 30 min at approximately 25 °C) as pretreatment protocols before hot-air drying of whole chokeberries at 50, 60 and 70 °C. Untreated and pretreated groups were dried for equal durations within each temperature. The study examined whether the kinetic responses varied with drying temperature, whether extending the plasma exposure from 150 to 300 s altered these responses in the same direction at every temperature, and how product attributes differed after the same drying duration. Drying kinetics were characterised using thin-layer models, apparent effective moisture diffusivity and apparent Arrhenius activation energy. Physical properties (shrinkage, water activity, rehydration, colour and browning), total phenolic, flavonoid and anthocyanin contents and in vitro antioxidant capacity (DPPH and CUPRAC) were assessed, with freeze-dried fruit from the same lot as the compositional reference. Surface microstructure was examined at 60 °C. The study provides a protocol-specific comparison of these pretreatments under a fixed-duration design; energy matching, process optimisation and mechanistic validation were outside its scope.

2. Materials and Methods

2.1. Raw Material

Black chokeberry fruits [*Aronia melanocarpa* (Michx.) Elliott, cv. 'Viking'] were obtained from a single harvest lot of a commercial orchard in Gebze (Kocaeli, Türkiye). The nine drying sessions were completed within approximately three weeks after harvest, with fruits stored at 4 °C until their respective experimental sessions. Intact fruits of similar size (mass 1.14 ± 0.07 g, equatorial diameter 12.0 ± 0.8 mm and polar height 11.0 ± 0.7 mm) were rinsed with distilled water and drained. The initial moisture content was 75.36 ± 0.66% on a wet basis (wb), determined gravimetrically at 105 °C to constant mass. Approximately 30 g of fruit (about 26 berries) was used per drying run.

Three independent portions of the same lot were frozen at −24 °C for 72 h and then transferred to a freeze-dryer (TRST-DS-4-4, Teknosem, İstanbul, Türkiye). They were dried in separate cycles at −55 °C and 10^{-3} mbar to provide the compositional reference (FD; powder moisture content 3.1% wb). Each cycle provided one independent FD replicate. Reagents and standards were of analytical grade (Sigma-Aldrich, St. Louis, MO, USA).

2.2. Pretreatments

Four groups were compared at each drying temperature: untreated control (Control), CP for 150 s (CP2.5) or 300 s (CP5), and indirect ultrasound (IUS). Fruits were transferred to the dryer immediately after pretreatment and, for IUS, after draining and blotting.

For CP treatment (Figure 1), approximately 30 g of fruit was arranged in a single layer in glass Petri dishes, 80 mm below the nozzle of an atmospheric-pressure plasma system (A5, Plasmatek, Isparta, Türkiye; 40 kHz; nominal power rating 600 W). Ambient air was supplied as the working gas at a flow rate of 9.42 L min^{-1}. The humidity and temperature of the gas at the nozzle, the power dissipated in the discharge and the composition and extent of the effluent were not measured. Exposure comprised successive 75 s cycles totalling 150 or 300 s, with manual turning of the fruit between cycles. The durations were chosen with reference to the minute-scale treatments previously applied to chokeberry for whole-fruit decontamination (1–3 min) [22] and dried-powder extraction (2 and 5 min) [23]; shorter or longer exposures were not tested. Fruit surface temperature was monitored during treatment with an infrared thermometer (830-T1, Testo SE & Co. KGaA, Titisee-Neustadt, Germany) and remained below 35 °C in both groups. The two plasma groups were treated

at the same device setting and differed only in exposure time. The 600 W value denotes the nominal device rating, not the power dissipated in the discharge or absorbed by the fruit. The Plasmatek A5 has previously been described as an atmospheric cold plasma system operating at 40 kHz [23,27], with an ambient-air flow rate of 9.42 L min^{-1} reported by Uzun [27]. The designation "cold plasma" follows these published descriptions. The fruit surface temperature characterises the heating of the fruit, not the temperature of the plasma gas.

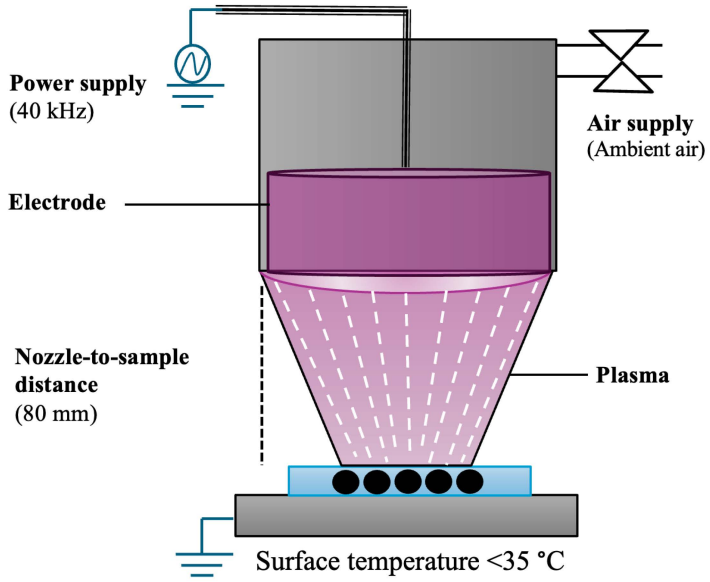


Figure 1. Schematic of the atmospheric-pressure cold plasma (CP) system used for the pretreatment of whole black chokeberry fruits.

IUS was applied in an ultrasonic bath (WUC-A06H, Daihan Scientific Co., Ltd., Wonju, Republic of Korea; 40 kHz; high-frequency peak output 168 W; tank 290 × 150 × 150 mm, 6 L). Whole fruits (approximately 30 g) were immersed in 120 mL of distilled water in a glass beaker (1:4, w/v) placed centrally in the bath, so that the acoustic energy reached the fruit through the bath water and the beaker wall without contact between the fruit and the transducer; this configuration is referred to here as indirect ultrasound. A comparable arrangement, a glass beaker placed centrally in the bath at a sample-to-water ratio of 1:4, has been described as indirect sonication for red bell pepper [28], and a beaker of water placed in a bath has also been used for yam slices [26]. The fruit-to-liquid ratio is close to the 1:5 (w/v) used for whole chokeberries in isotonic mannitol solution [5] and for whole myrtle berries in water or grape molasses [29]. Sonication was applied for 30 min, and the temperature of the ultrasonic bath water was monitored using a thermometer and was approximately 25 °C during treatment. The 30 min duration is consistent with two previous ultrasound studies on whole chokeberries [4,5], of which the first used a bath at 50 °C before vacuum drying and the second applied ultrasound at 25 °C. The bath operated at its fixed frequency of 40 kHz without programmed pulsing. Only one ultrasound frequency, treatment duration, fruit loading and operating mode were investigated; alternative conditions were not evaluated. The acoustic power delivered to the fruit was not measured. The fruits were then drained for 2 min, gently blotted and weighed, and the post-treatment mass was used to calculate the initial moisture content (Section 2.4.1).

2.3. Hot-Air Drying

Drying was performed at 50, 60 and 70 °C in four forced-convection ovens of the same make and model (UF110, Memmert GmbH, Schwabach, Germany). In each drying session, the four ovens were set to the same target temperature, and the Control, CP2.5, CP5 and IUS groups were dried in parallel, one group per oven. The assignment of the groups to the ovens was changed between replicates. Air velocity over the trays was 1.3 m s^{-1} in all four ovens, measured with a Testo 440 anemometer (Testo SE & Co. KGaA, Titisee-Neustadt, Germany). Relative humidity in the chambers was neither controlled nor monitored; ambient laboratory conditions were 22 ± 2 °C and 50 ± 5% RH. For each run, approximately 30 g of fruit was distributed in a single layer on a perforated stainless-steel tray (100 mm × 100 mm), a loading density of 3.0 kg m^{-2}.

Mass was recorded to the nearest 0.01 g with an analytical balance (ABS 80-4N, Kern & Sohn GmbH, Balingen, Germany) every 15 min during the first 120 min and approximately every 30 min thereafter, except for one 6–8 h overnight interval in the 50 and 60 °C runs, during which drying continued without weighing. Drying time was counted from the moment each sample was placed in the oven, and fixed durations of 35.5, 23.5 and 14.5 h were applied at 50, 60 and 70 °C, respectively. These durations were established in preliminary trials as the times at which untreated fruit fell below 10% moisture (wb), calculated from the sample mass and its dry-matter content (Section 2.4.1). All four groups received the same duration at each temperature, so the design equalised drying time among the pretreatment groups rather than the final moisture content. At each temperature, three drying sessions were started on different days, each with a new portion of fruit from the same harvest lot. The 36 drying runs were thus completed in nine sessions, with three replicates of each pretreatment × temperature combination ($n = 3$).

2.4. Drying Kinetics

2.4.1. Moisture Ratio and Drying Rate

For each run, the dry-matter fraction was determined from a representative subsample taken immediately after pretreatment and dried at 105 °C to constant mass and multiplied by the batch mass to obtain the dry-matter mass (m_d). The dry-basis moisture content (X) and the moisture ratio (MR) were calculated as

$$X_t = (m_t - m_d)/m_d \quad (1)$$

$$\mathrm{MR} = (X_t - X_e)/(X_0 - X_e) \quad (2)$$

where m_t is the sample mass at time t (g), and X_0 and X_e are the initial and equilibrium moisture contents (g/g dry basis, db). X_0 was obtained from Equation (1) at $t = 0$, using the post-treatment mass for the IUS group. X_e was determined for each run in a parallel sample that received the same pretreatment and drying temperature and was dried until three successive mass readings at 30 min intervals differed by less than 0.01 g; X_e was calculated with the dry-matter mass of that sample. The experimental samples were removed at the fixed duration and retained for the quality analyses. X_e ranged from 0.022 to 0.050 g g^{-1} db across the 36 runs, below the moisture content at the end of the fixed duration (0.057–0.112 g g^{-1} db), so MR at the end of each run was 0.007–0.026; the MR = 0.10 threshold used for t_{10} is therefore distinct from the 10% (wb) end point of the preliminary trials.

The drying rate was calculated as DR = $(X_t - X_{t+\Delta t})/\Delta t$ (g water g^{-1} dry matter min^{-1}), and each interval-averaged rate was assigned to the moisture content at the start of its weighing interval. For the comparison of pretreatments at matched moisture contents, the rates of each run were interpolated onto a common grid from 0.5 to 2.5 g g^{-1} dry matter at 0.1 g g^{-1} intervals. The mean of the interpolated rates was used as the run-level response

in the analysis of variance, and condition means were expressed relative to the control at the same temperature. The times to MR = 0.50 (t_{50}) and MR = 0.10 (t_{10}) and the time at which the moisture content fell to 40% (wb) ($t_{40\%\ wb}$) were obtained by linear interpolation between consecutive weighings, independently of the fitted models. Where a threshold fell within the overnight interval (eight of the twelve 50 °C runs for t_{10}), the interpolated time depends on the trajectory assumed between the two weighings; if MR decreases convexly across the interval, as the fitted models indicate, linear interpolation overestimates the time. The size of this effect was examined by solving the Midilli–Küçük equation fitted to each run (Section 2.4.2) for MR = 0.10 and comparing the model-based threshold times with the interpolated values (Section 3.1); the interpolated values are reported throughout.

2.4.2. Thin-Layer Model Fitting

The MR–time data of each run (34 to 65 observations) were fitted separately by nonlinear least squares using the unconstrained Levenberg–Marquardt algorithm (NLR procedure, IBM SPSS Statistics 26.0); all 144 fits converged, and the starting values and convergence criteria are given in the footnote to Supplementary Table S1. Four thin-layer models used in fruit drying [30] were fitted: the Henderson–Pabis model (3); the Page model (4); the Logarithmic model (5) [31]; and the Midilli–Küçük model (6) [32]:

$$\mathrm{MR} = a\,\exp(-kt) \tag{3}$$

$$\mathrm{MR} = \exp(-kt^n) \tag{4}$$

$$\mathrm{MR} = a\,\exp(-kt) + c \tag{5}$$

$$\mathrm{MR} = a\,\exp(-kt^n) + bt \tag{6}$$

where t is the drying time (min); a, c and n are dimensionless; b is in min^{-1}; and k is in min^{-1} for the Henderson–Pabis and Logarithmic models and min^{-n} for the Page and Midilli–Küçük models. The models were restricted to the observed MR range and their constants were treated as empirical descriptors. Because the Page model is obtained from the Midilli–Küçük model when $a = 1$ and $b = 0$, the improvement in fit between them was tested for each run with the extra sum-of-squares F-test for nested models ($p < 0.05$) [33].

Goodness of fit was evaluated with the coefficient of determination (R^2), the root mean square error (RMSE) and the reduced chi-square (χ^2), interpreted together with model complexity [34]. A higher R^2 and lower RMSE and χ^2 indicated closer agreement with the data, and any disagreement among the three criteria is reported. The statistics were calculated as

$$\mathrm{RMSE} = [\Sigma(\mathrm{MR_{pre,i}} - \mathrm{MR_{exp,i}})^2/\mathrm{N}]^{0.5} \tag{7}$$

$$\chi^2 = \Sigma(\mathrm{MR_{exp,i}} - \mathrm{MR_{pre,i}})^2/(\mathrm{N} - \mathrm{z}) \tag{8}$$

where $\mathrm{MR_{exp,i}}$ and $\mathrm{MR_{pre,i}}$ are the experimental and predicted moisture ratios, N is the number of observations and z the number of model constants. Model constants and fit statistics are reported as mean ± standard deviation (SD) of the three runs of each condition.

2.4.3. Calculation of Shrinkage, Apparent Effective Moisture Diffusivity and Activation Energy

The equatorial diameter (D_{eq}) and polar height (H) of every fruit were measured before and after drying with a digital calliper (CD-15AX, Mitutoyo Corp., Kawasaki, Japan; 0.01 mm). Fruit volume was approximated as an oblate spheroid, $V = \pi {D_{eq}}^2\mathrm{H}/6$ [35], and volumetric shrinkage was calculated from the paired initial and final volumes of each fruit as

$$\mathrm{S_V}\ (\%) = 100\ (V_0 - V_{end})/V_0 \tag{9}$$

where V_0 and V_{end} are the initial and final volumes. Individual values were averaged to give one S_V value per run.

The apparent effective moisture diffusivity (D_{eff}) was estimated from the first term of the analytical solution of Fick's second law for a sphere [36], assuming uniform initial moisture, constant diffusivity and negligible external resistance:

$$\ln(MR) = \ln(6/\pi^2) - (\pi^2 D_{eff} t)/r^2 \quad (10)$$

where t is the drying time (s) and r the characteristic radius (m). ln(MR) was regressed linearly on t within $0.20 \leq MR \leq 0.80$, where the relationship was closely linear (11 to 25 observations per run, $R^2 \geq 0.997$), with the intercept estimated freely rather than fixed at $\ln(6/\pi^2)$; D_{eff} was obtained from the slope, and the fitted intercepts are examined in Section 3.4. A common initial volume-equivalent radius, $r_0 = (D_{eq}{}^2H)^{1/3}/2 = 5.83$ mm, was calculated from the lot-mean dimensions in Section 2.1 and used for all runs, following the spherical approximation applied to whole grapes by Miraei Ashtiani et al. [17]. Measured shrinkage was not incorporated, and the estimates were treated as apparent, comparative transport parameters. Fruit dimensions were measured only before and after drying, so the course of shrinkage during drying is not known and no shrinkage term could be included in the calculation. Sensitivity to the fitting window was assessed by repeating the regressions over $0.20 \leq MR \leq 0.60$ with the same radius and free intercept (Supplementary Table S2).

The temperature dependence of D_{eff} was described by an Arrhenius-type relationship:

$$D_{eff} = D_0 \exp(-E_a/RT) \quad (11)$$

where D_0 is the pre-exponential factor ($m^2\ s^{-1}$), E_a the activation energy (kJ mol^{-1}), R the universal gas constant (0.008314 kJ $mol^{-1}\ K^{-1}$) and T the absolute drying temperature (K). For each pretreatment, $\ln(D_{eff})$ was regressed on $1/T$ using the three independent estimates at each of the three temperatures (nine values). The value obtained is an apparent activation energy describing the temperature sensitivity of the apparent diffusivity between 50 and 70 °C; it is not a measure of the energy required for drying, which was not determined.

2.5. Rehydration

Whole dried fruits (5.0 ± 0.1 g) were immersed in distilled water (1:20, w/v) in a covered beaker at 25 °C for 24 h, following the immersion procedure of Demircan et al. [6], then drained on a sieve for 2–3 min, blotted and weighed. The rehydration ratio (RR) was calculated as the rehydrated mass divided by the dried-sample mass, and the rehydrated mass was also expressed per unit dry-matter mass of the dried sample to account for the different residual moisture contents.

2.6. Sample Preparation, Water Activity, Colour and Browning Indices

After cooling, the remaining fruits of each run were milled with a blade grinder (Simbo, İstanbul, Türkiye), passed through a 2.0 mm stainless-steel sieve (Retsch GmbH, Haan, Germany) and stored in light-protected airtight polypropylene containers at 4 °C. The analyses were completed within two weeks. Compositional results are expressed on a dry-matter (DM) basis using the moisture content of each powder, determined at 105 °C to constant mass.

Water activity (a_w) was measured at 25.0 ± 0.2 °C with a calibrated water activity meter (LabTouch-aw, Novasina AG, Lachen, Switzerland). Powder colour was measured with a colorimeter (CR-400, Konica Minolta Sensing Inc., Osaka, Japan; illuminant D65, 2° observer); L*, a* and b* denote lightness, redness–greenness and yellowness–blueness. Five positions were measured per batch and averaged, so that the reported values are means of three batches ($n = 3$). The hue angle (h°) was calculated according to Pathare et al. [37]

and is reported as a signed angle relative to the +a* axis, because the values lie on either side of 0°.

Two browning indices were determined. The CIELAB browning index, BI(CIELAB), was calculated from L*, a* and b* [38] as

$$\mathrm{BI(CIELAB)} = 100\ (x - 0.31)/0.172 \quad (12)$$

$$x = (a^* + 1.75\ L^*)/(5.645\ L^* + a^* - 3.012\ b^*) \quad (13)$$

The spectrophotometric browning index, BI(A420), was determined according to Demircan et al. [6]. Powder (0.50 g) was homogenised with 5 mL of 2% acetic acid and held at 4 °C for 30 min; after mixing for 3 min, the suspension was centrifuged at 3500 rpm for 10 min, and the sediment was re-extracted with 2.5 mL of 2% acetic acid and centrifuged again. The combined supernatants were mixed with 1 mL of 10% (*w*/*v*) lead acetate, made up to 20 mL with 2% acetic acid and centrifuged at 3500 rpm for 5 min; the supernatant was then mixed with an equal volume of absolute ethanol and centrifuged once more. Absorbance was read at 420 and 600 nm against a 1:1 (*v*/*v*) mixture of 2% acetic acid and ethanol, and BI(A420) was calculated from the turbidity-corrected difference $A_{420} - A_{600}$ with correction for sample mass and dilution. Triplicate readings of each extract were averaged. BI(A420) is expressed as absorbance units per g of powder as analysed and was not corrected for the moisture content of the powder.

Water activity and colour were measured on the powders as such. TPC, TFC, TMA and cupric ion reducing antioxidant capacity (CUPRAC) (Sections 2.7–2.9) are expressed per unit dry matter using the moisture content of each powder; 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical-scavenging activity is reported as percentage inhibition at a fixed extract dilution and is not expressed on a dry-matter basis; the rehydration ratio is given per unit dried mass and per unit dry matter (Section 2.5).

2.7. Total Phenolic and Total Flavonoid Contents

Powder (0.10 g) was mixed with 10.0 mL of 80% (*v*/*v*) ethanol containing 0.1% (*v*/*v*) HCl, vortexed and centrifuged at 5000 rpm for 10 min at 25 °C [6]. Extraction was repeated until the supernatant was clear, and the combined supernatants were made up to 25.0 mL with the extraction solvent (4 mg powder mL^{-1}) and stored at −18 °C until analysis. The FD reference was extracted and analysed in the same way.

Total phenolic content (TPC) was determined by the Folin–Ciocalteu method [39]. Extract (0.10 mL) was mixed with 5.0 mL of 0.1 M Folin–Ciocalteu reagent and 4.0 mL of 7.5% (*w*/*v*) Na_2CO_3, vortexed for 2 min and held at room temperature in the dark for 1 h, and the absorbance was read at 760 nm. TPC was calculated from a gallic acid calibration curve and expressed as mg gallic acid equivalents per 100 g DM (mg GAE/100 g DM).

Total flavonoid content (TFC) was determined by the aluminium chloride method [40]. Extract (0.50 mL), 2.0 mL of water and 0.15 mL of 5% (*w*/*v*) $NaNO_2$ were mixed; after 6 min, 0.15 mL of 10% (*w*/*v*) $AlCl_3$ was added, and after a further 6 min, 2.0 mL of 4% (*w*/*v*) NaOH and 0.70 mL of water. The reaction proceeded at room temperature, and the absorbance was read at 510 nm after a further 15 min. TFC was calculated from a catechin calibration curve and expressed as mg catechin equivalents per 100 g DM (mg CE/100 g DM).

2.8. Total Monomeric Anthocyanins

Powder (0.20 g) was extracted to 10.0 mL with 96% ethanol–1.5 N HCl (85:15, *v*/*v*), centrifuged at 4500 rpm for 20 min, kept at 4 °C overnight and centrifuged again. Total monomeric anthocyanin content (TMA) was determined in the extract by the pH-differential method of Lee et al. [41]. Separate 1.0 mL aliquots were diluted to 10.0 mL with 0.025 M potassium chloride buffer (pH 1.0) and 0.4 M sodium acetate buffer (pH 4.5), held for

30 min in the dark, and the absorbance was read at 520 and 700 nm in 1 cm cuvettes against distilled water. TMA was calculated from the haze-corrected absorbance difference between the two pH conditions, $(A_{520} - A_{700})$pH 1.0 − $(A_{520} - A_{700})$pH 4.5, using a molecular mass of 449.2 g mol^{-1} and a molar absorptivity of 26,900 L mol^{-1} cm^{-1} for cyanidin-3-glucoside. Results were corrected for dilution, extraction volume and sample dry-matter mass and are expressed as mg cyanidin-3-glucoside equivalents (C3G) per 100 g DM.

2.9. Antioxidant Capacity

DPPH radical-scavenging activity was determined according to Brand-Williams et al. [42]. Extract prepared as described in Section 2.7 (0.15 mL) was mixed with 2.85 mL of 1.1×10^{-4} M methanolic DPPH, held for 30 min at room temperature in the dark and read at 515 nm. This corresponds to 0.60 mg of powder per reaction (0.20 mg powder mL^{-1} in the reaction mixture); the assay was standardised to powder mass, not to equal dry-matter mass. Inhibition (%) was calculated as 100 $(A_{control} - A_{sample})/A_{control}$, where $A_{control}$ and A_{sample} are the absorbances of the assay control and of the sample reaction. The FD extract was measured at a two-fold dilution, so its inhibition values were not compared directly with those of the hot-air-dried samples.

CUPRAC was determined according to Apak et al. [43]. Extract (0.10 mL) and water (0.90 mL) were combined with 3.0 mL of a reagent prepared from equal volumes of 10 mM $CuCl_2$, 7.5 mM neocuproine in 96% ethanol and 1.0 M ammonium acetate buffer (pH 7.0), held for 60 min in the dark and read at 450 nm. Results are expressed as mg Trolox equivalents per g DM (mg TE/g DM).

2.10. Microstructure

The surface of dried fruits from the four groups dried at 60 °C was examined by scanning electron microscopy with an EVO LS 50 microscope (Carl Zeiss AG, Feldbach, Switzerland). Whole fruits were mounted on aluminium stubs, sputter-coated with gold and imaged with a secondary electron detector at 10 kV and 200× magnification. One representative field per group is presented; arrows indicate features of interest, and brightness and contrast were not adjusted. The images were used qualitatively, without morphometric analysis.

2.11. Statistical Analysis

The independent drying run was the experimental unit ($n = 3$ per pretreatment × temperature condition), and measurements repeated within a run were averaged before analysis. Results are reported as mean ± SD. The effects of pretreatment, drying temperature and their interaction were tested by two-way analysis of variance (ANOVA) on the 4 × 3 design. Planned simple-effect comparisons were made by one-way ANOVA followed by Tukey's HSD test ($p < 0.05$), comparing pretreatments within each drying temperature (lowercase letters in the tables) and drying temperatures within each pretreatment (uppercase letters); when the interaction was significant, interpretation focused on the simple effects, and otherwise on the main effects. The relation between final moisture content and volumetric shrinkage was examined by Pearson correlation across the 36 runs and within each temperature ($n = 12$).

TPC, TFC, TMA and CUPRAC of the twelve hot-air-dried conditions were compared with the FD reference by one-way ANOVA followed by Dunnett's test ($p < 0.05$). Differences among the Arrhenius slopes were tested as the pretreatment × $(1/T)$ interaction in an analysis of covariance (ANCOVA) of $\ln(D_{eff})$. All analyses were performed with IBM SPSS Statistics 26.0 (IBM Corp., Armonk, NY, USA).

3. Results and Discussion

3.1. Drying Curves and Characteristic Drying Times

The drying curves of the four groups at 50, 60 and 70 °C are shown in Figure 2A–D. Initial moisture content did not differ among the four groups at any temperature ($p \geq 0.064$). With the drying duration fixed within each temperature, the pretreatment differences were reflected in the final moisture content (Table 1): untreated fruit finished at 8.6–9.9% (wb) and all pretreated groups below the control (5.6–7.7% wb; $p < 0.05$), with CP2.5 giving the lowest mean at each temperature.

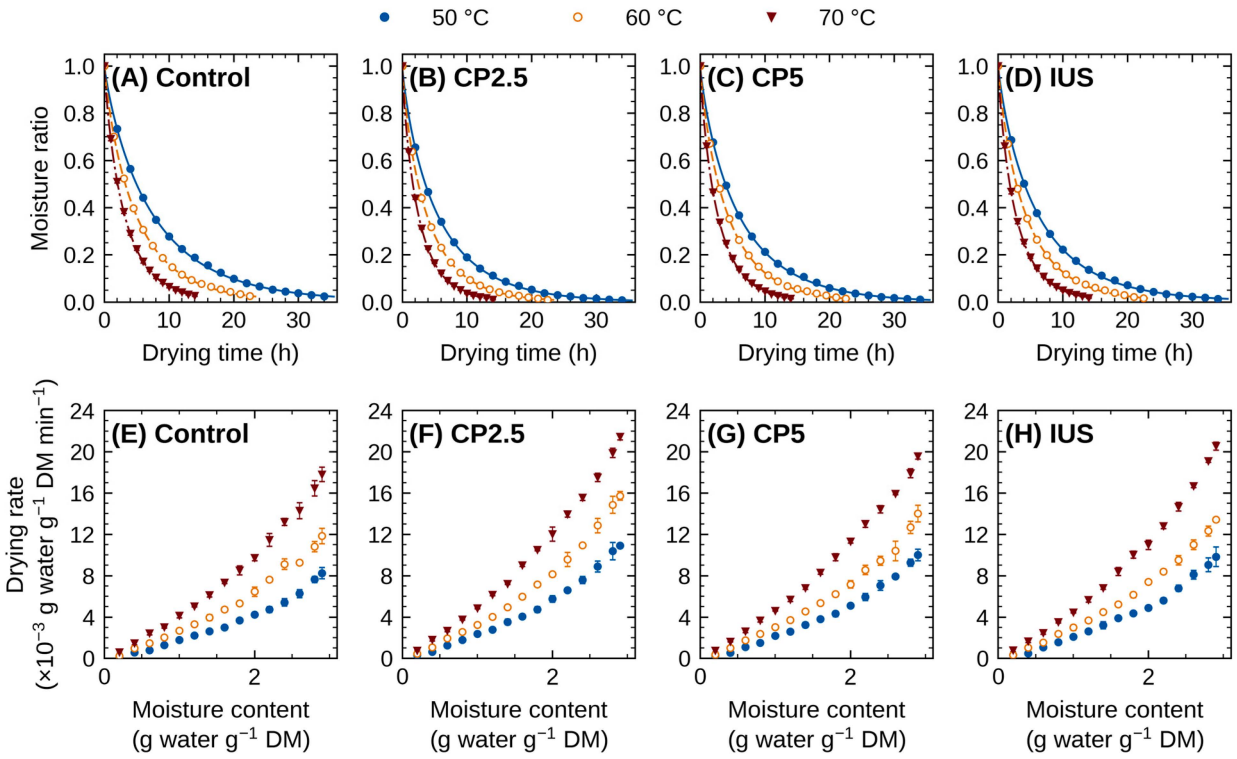


Figure 2. Moisture-ratio curves (**A**–**D**) and drying-rate curves (**E**–**H**) of whole black chokeberry fruits dried at 50, 60 and 70 °C without pretreatment (Control; (**A**,**E**)), after cold plasma for 150 s (CP2.5; (**B**,**F**)) or 300 s (CP5; (**C**,**G**)), or after indirect ultrasound for 30 min (IUS; (**D**,**H**)). Symbols are means of three independent drying runs and error bars show the between-run SD. In (**A**–**D**) the SD did not exceed 0.013 MR units at any point and is hidden by the symbols; in (**E**–**H**) the error bars show the SD of the drying rate. For clarity, every second point of the common time grid is displayed in (**A**–**D**), and every second point of the common moisture grid, together with the final point, in (**E**–**H**). Lines in (**A**–**D**) are Midilli–Küçük curves generated from the mean parameter estimates for each condition (Section 3.3); no lines are drawn in (**E**–**H**). Drying rate is expressed per unit dry matter (DM), and drying proceeds from right to left as moisture content decreases. The matched-moisture statistical analysis used 0.5–2.5 g water g^{-1} DM (Section 2.4.1).

The characteristic drying times showed the same treatment pattern. In untreated fruit, t_{10} fell from 19.82 h at 50 °C to 8.19 h at 70 °C, a 59% reduction, larger than that achieved by any pretreatment within a temperature. Every pretreatment shortened t_{50}, t_{10} and $t_{40\%\,wb}$ at every temperature ($p < 0.05$): relative to the control, t_{10} was reduced by 18.8–25.1% with CP2.5, 12.4–16.7% with CP5 and 11.0–13.1% with IUS. CP2.5 gave shorter characteristic times than CP5 at every temperature, whereas CP5 and IUS differed only in t_{50} at 50 °C. The pretreatment × temperature interaction was significant for all three characteristic times ($p < 0.001$); for CP2.5 the reduction in t_{10} fell from 4.97 h (25%) at 50 °C to 1.54 h (19%) at 70 °C. The interpolated reductions in t_{10} at 50 °C were 0.7–3.1 percentage points smaller

than those obtained by solving the Midilli–Küçük equation fitted to each run (25.8, 19.8 and 16.2% for CP2.5, CP5 and IUS), and the treatment ordering was the same; at 60 and 70 °C the two methods differed by at most 0.6 percentage points (Section 2.4.1).

Table 1. Final moisture content, characteristic drying times, volumetric shrinkage and apparent effective moisture diffusivity of whole black chokeberry fruits dried at 50, 60 and 70 °C after cold plasma (CP2.5, CP5) and indirect ultrasound (IUS) pretreatments.

T (°C)	Pretreatment	Final Moisture (% wb)	t_{50} (h)	t_{10} (h)	$t_{40\%\ wb}$ (h)	S_V (%)	D_{eff} ($\times 10^{-10}$ m^2 s^{-1})
50	Control	9.9 ± 0.2 ^{a}	4.95 ± 0.06 ^{a}	19.82 ± 0.24 ^{a}	12.72 ± 0.24 ^{a}	50.2 ± 0.4 ^{b}	1.14 ± 0.01 ^{d}
	CP2.5	6.2 ± 0.2 ^{c}	3.59 ± 0.03 ^{d}	14.85 ± 0.51 ^{c}	9.39 ± 0.30 ^{c}	46.2 ± 0.6 ^{c}	1.53 ± 0.02 ^{a}
	CP5	6.8 ± 0.5 ^{bc}	3.92 ± 0.03 ^{c}	16.52 ± 0.16 ^{b}	10.24 ± 0.17 ^{b}	43.9 ± 0.4 ^{d}	1.40 ± 0.01 ^{b}
	IUS	7.7 ± 0.6 ^{b}	4.02 ± 0.02 ^{b}	17.23 ± 0.32 ^{b}	10.54 ± 0.47 ^{b}	55.5 ± 0.4 ^{a}	1.36 ± 0.01 ^{c}
60	Control	8.9 ± 0.3 ^{a}	3.25 ± 0.01 ^{a}	12.99 ± 0.06 ^{a}	8.29 ± 0.31 ^{a}	50.7 ± 0.4 ^{b}	1.73 ± 0.01 ^{c}
	CP2.5	5.6 ± 0.3 ^{c}	2.48 ± 0.06 ^{c}	10.18 ± 0.05 ^{c}	6.30 ± 0.16 ^{c}	47.0 ± 1.0 ^{c}	2.20 ± 0.02 ^{a}
	CP5	6.6 ± 0.2 ^{b}	2.82 ± 0.05 ^{b}	11.18 ± 0.13 ^{b}	7.23 ± 0.29 ^{b}	43.0 ± 0.9 ^{d}	1.98 ± 0.02 ^{b}
	IUS	7.1 ± 0.5 ^{b}	2.82 ± 0.12 ^{b}	11.48 ± 0.39 ^{b}	7.24 ± 0.14 ^{b}	54.8 ± 1.1 ^{a}	1.98 ± 0.07 ^{b}
70	Control	8.6 ± 0.7 ^{a}	2.07 ± 0.04 ^{a}	8.19 ± 0.22 ^{a}	5.25 ± 0.16 ^{a}	49.4 ± 1.4 ^{b}	2.72 ± 0.04 ^{c}
	CP2.5	5.7 ± 0.2 ^{c}	1.64 ± 0.02 ^{c}	6.65 ± 0.08 ^{c}	4.35 ± 0.03 ^{c}	47.2 ± 0.3 ^{b}	3.37 ± 0.03 ^{a}
	CP5	6.2 ± 0.4 ^{bc}	1.79 ± 0.02 ^{b}	7.17 ± 0.08 ^{b}	4.64 ± 0.04 ^{b}	44.3 ± 0.9 ^{c}	3.12 ± 0.04 ^{b}
	IUS	6.9 ± 0.1 ^{b}	1.79 ± 0.05 ^{b}	7.29 ± 0.14 ^{b}	4.61 ± 0.06 ^{b}	54.9 ± 0.8 ^{a}	3.07 ± 0.06 ^{b}

Values are mean ± SD (n = 3). Different lowercase letters within a drying temperature indicate significant differences among pretreatments (Tukey's HSD, $p < 0.05$). Final moisture content is the value reached at the end of the fixed drying duration (35.5, 23.5 and 14.5 h at 50, 60 and 70 °C). t_{50} and t_{10}, times to MR = 0.50 and MR = 0.10; $t_{40\%\ wb}$, time at which the moisture content fell to 40% (wb); all three were obtained by linear interpolation between consecutive weighings, and at 50 °C t_{10} fell within the overnight weighing interval in eight of the twelve runs (Section 2.4.1); S_V, volumetric shrinkage; D_{eff}, apparent effective moisture diffusivity calculated over 0.20 ≤ MR ≤ 0.80 from the ln(MR)–time slope with the initial volume-equivalent radius (r_0 = 5.83 mm) held constant; it is an apparent, slope-derived value that does not allow for shrinkage.

The longer plasma exposure gave the smaller kinetic benefit at every temperature and for all three characteristic times, although the two plasma groups differed in final moisture content only at 60 °C (Table 1). The dependence of the drying response on plasma exposure time has differed among studies. In whole grapes, extending gliding arc treatment from 50 to 60 s did not shorten hot-air drying further, which the authors attributed tentatively to the formation of a surface crust and noted would need to be confirmed [17]. In jujube slices dried at 70 °C, the effective diffusivity rose from 3.79 × 10^{-10} m^2 s^{-1} in the control to 6.76 × 10^{-10} m^2 s^{-1} after 15 s of plasma jet treatment per side and then fell to 4.26 and 3.25 × 10^{-10} m^2 s^{-1} after 30 and 60 s per side, while the surface temperature rose from 25.7 °C in untreated slices to 54.2 °C after the longest treatment [18]. In apple slices dried by vacuum microwave, by contrast, drying time decreased from 48 to 36 min as dielectric barrier discharge exposure was extended from 0 to 80 s, whereas the authors reported weaker retention of individual phenolic compounds at 80 s than at 20–60 s [38]. In apple slices dried by hot air, 60 and 90 s of low-pressure plasma shortened drying by 33.3 and 11.1%, whereas 120 s did not differ significantly from the untreated control [11]. The response to plasma exposure time therefore appears to depend on the matrix, the plasma source and the drying process [10], and the present results apply to whole chokeberry under the conditions tested. The two plasma groups were treated at the same nominal device setting, with only the exposure time varied, and the fruit surface remained below 35 °C in both; the characterisation available (Section 2.2) and the post-drying micrographs (Section 3.7) do not show why CP5 gave the smaller kinetic benefit. In whole blueberries dried at 65 °C, diffusivity increased with both exposure time (6–18 s) and power (300–900 W) [19], in a design in which the two variables were varied separately. Here only the exposure time was

varied. With two exposure times, the study cannot locate an optimum. It shows only that the shorter exposure gave the larger kinetic response.

The 11–13% reduction in t_{10} with IUS fell below the 21.9 and 22.9% reported at 65 and 70 °C for whole chokeberry sonicated for the same time at a higher nominal power, and was similar in magnitude to the 11.8% reported at 75 °C [5]. In that study, the fruit was sonicated directly in isotonic mannitol solution and drying was followed to a moisture ratio of 0.05 rather than 0.10. The effect of sonication duration has also been examined in chokeberries sonicated in an ultrasonic bath and subsequently dried as halves under infrared heating: the shortest drying times were obtained after 10 min of sonication, and longer treatments increased drying time at 60 °C (15 and 20 min) and at 70 °C (20 min), with no change at 80 °C [14]. Since the fruit preparation and the drying method differed from those used here, these findings do not indicate an optimal duration for hot-air drying of whole chokeberries. The single 30 min treatment used here was not compared with shorter or longer durations. Because an immersion-only control was not included, the IUS response represents the combined effect of the sonication and the 30 min of water immersion.

3.2. Drying Rate

The drying rate declined continuously with moisture content and no constant-rate period was apparent (Figure 2E–H), as also reported for whole blueberries dried in hot air [44]. In all 36 runs, the highest rate was that of the first 15 min interval, and in untreated fruit this initial interval-averaged rate rose from 8.84×10^{-3} to 20.20×10^{-3} g water g^{-1} DM min^{-1} between 50 and 70 °C. Whole chokeberry dried at a lower air velocity (0.25 m s^{-1}) showed the same direction of temperature dependence, although the maximum mass-loss rate was reported in g h^{-1} and later in the drying curve [15].

At matched moisture contents, the run-level mean drying rate over 0.5–2.5 g g^{-1} DM differed among pretreatments ($F(3,24) = 236.0$, $p < 0.001$) and temperatures ($F(2,24) = 5590.4$, $p < 0.001$), with a significant interaction ($F(6,24) = 3.90$, $p = 0.007$). The ranking was consistent across temperatures: CP2.5 gave the highest mean rate, CP5 and IUS were intermediate and did not differ from each other ($p \geq 0.13$), and the control was lowest ($p \leq 0.003$ for all other pairwise comparisons). Relative to the control, the mean rate was higher by 21–36% for CP2.5, 12–26% for CP5 and 12–22% for IUS, with the largest relative increases at 50 °C. For CP2.5 the absolute increase rose with temperature, from 1.08×10^{-3} to 1.48×10^{-3} g water g^{-1} DM min^{-1}, whereas the relative increase declined from 36 to 21%, a pattern consistent with the significant interaction. These higher rates indicate faster moisture removal under the conditions tested, but not which transport resistance controls it, since matching the mean moisture content does not equalise the surface state or the temperature of the fruit. Micron-scale openings have been reported in whole grape skin after gliding arc plasma treatment, although the surface of the present fruit was examined only after drying [17].

3.3. Thin-Layer Modelling

All four models described the drying curves closely, with R^2 above 0.995 in every fit, but not equally well: averaged across conditions, the RMSE of the Henderson–Pabis and Logarithmic models was six to ten times that of the Page and Midilli–Küçük models (Table 2; Supplementary Table S1). The Midilli–Küçük model gave the highest R^2 and the lowest RMSE in all 36 runs and the lowest reduced χ^2 in 35 of them, and the extra sum-of-squares F-test was significant in 24 ($p < 0.05$). That test assessed whether freeing the two constants jointly improved the fit, rather than the significance of either one. The improvement was small in absolute terms, so the Page model provided a close two-constant description of the data within the range studied, while the Midilli–Küçük model gave the closest overall fit.

Midilli-based models have also provided close fits for whole chokeberry dried under other convective conditions [5,16].

Table 2. Constants of the Midilli–Küçük model and goodness of fit of the four thin-layer models fitted to the drying curves of whole black chokeberry fruits.

T (°C)	Pretreatment	*a*	*k* ($\times 10^{-3}$ min^{-n})	*n*	*b* ($\times 10^{-6}$ min^{-1})	R^2 HP	R^2 Page	R^2 Log	R^2 MK	RMSE HP ($\times 10^{-3}$)	RMSE Page ($\times 10^{-3}$)	RMSE Log ($\times 10^{-3}$)	RMSE MK ($\times 10^{-3}$)
50	Control	1.003 ± 0.002	4.84 ± 0.14	0.872 ± 0.003	0.70 ± 0.46	0.998	>0.999	0.999	>0.999	14.55	1.87	8.94	1.55
	CP2.5	1.002 ± 0.001	7.23 ± 0.12	0.850 ± 0.001	−0.55 ± 0.03	0.997	>0.999	0.998	>0.999	14.24	1.44	11.76	1.34
	CP5	1.002 ± 0.001	6.49 ± 0.26	0.856 ± 0.007	−0.84 ± 0.52	0.998	>0.999	0.999	>0.999	13.75	1.60	10.93	1.40
	IUS	1.006 ± 0.002	6.45 ± 0.04	0.853 ± 0.002	0.56 ± 0.27	0.997	>0.999	0.998	>0.999	15.26	2.13	11.13	1.72
60	Control	1.007 ± 0.002	7.27 ± 0.09	0.866 ± 0.002	1.38 ± 0.84	0.997	>0.999	0.998	>0.999	15.96	2.87	11.14	2.38
	CP2.5	1.003 ± 0.001	9.69 ± 0.45	0.854 ± 0.006	−0.40 ± 0.39	0.997	>0.999	0.998	>0.999	16.54	1.80	12.76	1.68
	CP5	1.002 ± 0.001	8.11 ± 0.26	0.868 ± 0.004	−0.55 ± 0.53	0.997	>0.999	0.998	>0.999	14.94	1.44	11.36	1.38
	IUS	1.005 ± 0.001	8.47 ± 0.48	0.860 ± 0.005	0.93 ± 0.44	0.997	>0.999	0.998	>0.999	16.27	2.27	12.27	1.90
70	Control	1.002 ± 0.001	10.29 ± 0.34	0.874 ± 0.003	2.00 ± 1.71	0.997	>0.999	0.999	>0.999	15.32	1.74	10.25	1.49
	CP2.5	1.003 ± 0.001	13.43 ± 0.27	0.859 ± 0.003	−0.87 ± 0.79	0.997	>0.999	0.998	>0.999	15.99	1.56	12.18	1.46
	CP5	1.002 ± 0.001	11.85 ± 0.40	0.870 ± 0.005	−0.67 ± 1.29	0.997	>0.999	0.998	>0.999	14.90	1.48	11.06	1.38
	IUS	1.003 ± 0.001	12.28 ± 0.52	0.862 ± 0.005	2.07 ± 1.04	0.997	>0.999	0.998	>0.999	16.66	2.01	11.63	1.71

Constants are mean ± SD ($n = 3$); R^2 and RMSE are means of three replicate fits. R^2 is given to three decimal places, and values above 0.999 are shown as >0.999. HP, Henderson–Pabis; Log, Logarithmic; MK, Midilli–Küçük. The improvement in fit from the Page to the Midilli–Küçük model was significant (extra sum-of-squares F-test, $p < 0.05$) in 24 of the 36 drying runs. Constants of the other models are given in Supplementary Table S1.

For the Midilli–Küçük model, *k* increased 2.1-fold between 50 and 70 °C in untreated fruit and was higher in the pretreated groups at each temperature, by 31–49% for CP2.5 and by less in CP5 and IUS ($p < 0.05$, except CP5 at 60 °C; Table 2). CP2.5 differed from CP5 at every temperature ($p \leq 0.006$), whereas CP5 and IUS never differed ($p \geq 0.56$), and no significant pretreatment × temperature interaction was detected for *k* ($p = 0.085$). The exponent *n* lay in a narrow range, between 0.850 and 0.874; *a* remained close to unity and *b* was of the order of 10^{-6} min^{-1} with a sign that differed between groups (Table 2). In whole chokeberry sonicated before hot-air drying, the Midilli–Küçük *k* was instead lower than in the untreated control at all three temperatures even though drying was shorter, and *n* differed between the two groups [5]. Because *k* is expressed in min^{-n} and its value depends on *n*, values obtained with different exponents are not on a common scale, and these constants were treated as empirical descriptors of the curves rather than as independent physical transport parameters.

3.4. Shrinkage, Apparent Effective Moisture Diffusivity and Activation Energy

Volumetric shrinkage differed among pretreatments, whereas no significant effect of drying temperature ($p = 0.97$) and no pretreatment × temperature interaction ($p = 0.099$) were detected (Table 1). Untreated fruit lost about half of its initial volume (49–51%), the plasma groups less (CP2.5 46–47%, CP5 43–44%) and IUS more (55%), the four groups differing at every temperature ($p < 0.05$) except CP2.5 and the control at 70 °C. Lower shrinkage has also been reported for whole grapes after gliding arc plasma treatment [17].

Because the drying duration was fixed within each temperature, the groups also ended drying at different moisture contents (Table 1), and part of the shrinkage difference could reflect the final moisture rather than the pretreatment itself. Across the 36 runs, volumetric shrinkage was weakly correlated with final moisture content ($r = 0.37$, $p = 0.028$) but not within any temperature ($r = 0.28$–0.44, $p \geq 0.15$, $n = 12$). The treatment ranking in shrinkage (IUS > control > CP2.5 > CP5) did not consistently follow that in final moisture content (control > IUS > CP5 > CP2.5); however, the contributions of moisture history and of treatment-induced structural changes could not be separated. Dimensions were measured only before and after drying, and the course of shrinkage during drying was not followed.

The apparent D_{eff} of untreated fruit increased 2.4-fold from 1.14×10^{-10} m^2 s^{-1} at 50 °C to 2.72×10^{-10} m^2 s^{-1} at 70 °C, and all pretreatments raised D_{eff} at every temperature ($p < 0.05$; Table 1). Relative to the control, the increase was 23.7–34.0% for CP2.5, 14.6–22.9% for CP5 and 12.6–19.1% for IUS, largest at 50 °C in each case. CP2.5 gave the highest values at every temperature, whereas CP5 exceeded IUS only at 50 °C ($p = 0.019$), and the pretreatment × temperature interaction was significant ($p < 0.001$). An increase of about 30% in effective diffusivity has been reported for whole grapes after gliding arc plasma pretreatment [17], and a higher effective diffusivity after ultrasound pretreatment for whole chokeberry [5].

The condition-mean intercepts of the ln(MR)–time regressions, −0.090 to −0.108, were far from the theoretical first-term value of $\ln(6/\pi^2) = -0.498$. Across individual runs, narrowing the regression window from $0.20 \leq MR \leq 0.80$ to $0.20 \leq MR \leq 0.60$ lowered D_{eff} by 2.1–4.4% without altering the ranking of the pretreatments (Supplementary Table S2). Because a fixed initial radius was used and the measured shrinkage was not incorporated in the calculation, D_{eff} is an apparent, slope-derived coefficient used here to compare treatments rather than as an intrinsic diffusivity. Estimates obtained without allowing for shrinkage have been reported to overestimate diffusive transport [30].

The Arrhenius plots were closely described by linear regressions (R^2 0.991–0.997; Figure 3). The estimated apparent activation energy was 40.2 kJ mol^{-1} for the control and 36.5, 36.9 and 37.6 kJ mol^{-1} for CP2.5, CP5 and IUS, that is 2.6–3.7 kJ mol^{-1} lower in the pretreated groups. The differences among the Arrhenius slopes were not significant (analysis of covariance, pretreatment × $1/T$, $p = 0.099$), and each regression rests on only three temperature levels, so these values indicate a numerical tendency rather than a demonstrated change in the temperature sensitivity of moisture transport. These values describe the temperature sensitivity of the apparent diffusivity, not the energy required for drying (Section 2.4.3). Görgüç et al. [5] likewise reported a lower numerical activation energy after ultrasound pretreatment of whole chokeberry, from 61.9 to 51.1 kJ mol^{-1} under different drying conditions.

3.5. Water Activity, Rehydration and Colour

Table 3 gives the water activity and colour of the powders and the rehydration ratio of the corresponding whole dried fruits. The pattern in water activity paralleled that of the final moisture content, decreasing in the order control > IUS > CP5 > CP2.5, with all pretreatment differences significant within each temperature except CP2.5 versus CP5 at 70 °C. It also fell with drying temperature within each pretreatment ($p \leq 0.009$), with no significant pretreatment × temperature interaction ($p = 0.51$). The pretreated groups fell within the 0.17–0.33 range reported for chokeberry dried by other methods [45].

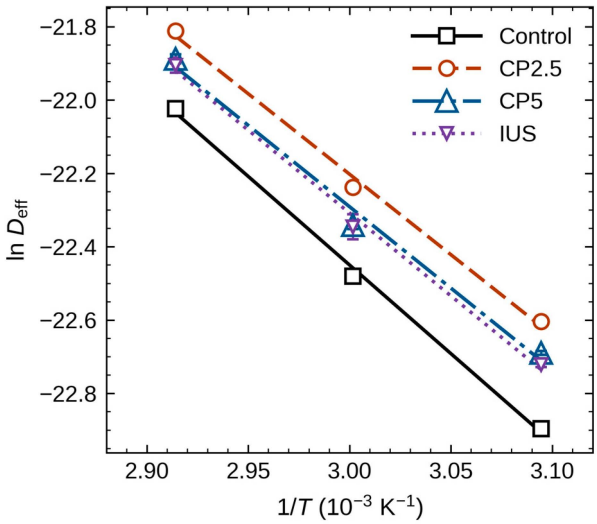


Figure 3. Arrhenius plots of the apparent effective moisture diffusivity (D_{eff}, m^2 s^{-1}) of whole black chokeberry fruits. Symbols represent means ± SD of three independent drying runs at each temperature. Lines are least-squares regressions of ln D_{eff} on $1/T$, fitted to the nine run-level D_{eff} estimates for each pretreatment. The slopes were used to estimate the activation energies reported in Section 3.4.

Table 3. Water activity, colour parameters and browning indices of the powders, and rehydration ratio of the whole dried fruits, of black chokeberry dried at 50, 60 and 70 °C after cold plasma (CP2.5, CP5) and indirect ultrasound (IUS) pretreatments.

T (°C)	Pretreatment	a_w	RR	RR_{DM}	L*	a*	b*	h°	BI (CIELAB)	BI (A420)
50	Control	0.397 ± 0.010 ^{aA}	2.01 ± 0.01 ^{bA}	2.23 ± 0.01 ^{bcA}	26.05 ± 0.73 ^{aB}	15.06 ± 0.10 ^{aA}	−3.36 ± 0.34 ^{bB}	−12.6 ± 1.2 ^{aB}	24.5 ± 0.5 ^{aB}	3.81 ± 0.21 ^{bB}
	CP2.5	0.264 ± 0.006 ^{dA}	2.19 ± 0.05 ^{aA}	2.33 ± 0.06 ^{abA}	27.73 ± 1.33 ^{aB}	11.46 ± 1.21 ^{bcA}	−5.48 ± 0.41 ^{cA}	−25.7 ± 3.0 ^{bA}	8.9 ± 3.2 ^{bA}	3.19 ± 0.02 ^{cA}
	CP5	0.298 ± 0.006 ^{cA}	2.23 ± 0.08 ^{aA}	2.40 ± 0.10 ^{aA}	26.28 ± 2.97 ^{aB}	10.21 ± 1.40 ^{cA}	−1.85 ± 0.71 ^{aA}	−10.7 ± 5.6 ^{aA}	19.4 ± 7.4 ^{abA}	3.00 ± 0.04 ^{cC}
	IUS	0.326 ± 0.009 ^{bA}	1.98 ± 0.05 ^{bA}	2.15 ± 0.04 ^{cA}	27.39 ± 0.02 ^{aB}	13.80 ± 0.38 ^{abA}	−1.33 ± 0.57 ^{aA}	−5.5 ± 2.2 ^{aA}	27.9 ± 1.3 ^{aA}	5.03 ± 0.16 ^{aA}
60	Control	0.367 ± 0.011 ^{aB}	2.01 ± 0.04 ^{bA}	2.21 ± 0.05 ^{bA}	32.34 ± 1.35 ^{aA}	12.89 ± 2.20 ^{aA}	−0.04 ± 0.94 ^{aA}	−0.6 ± 4.0 ^{aA}	26.2 ± 6.2 ^{aB}	3.72 ± 0.07 ^{aB}
	CP2.5	0.246 ± 0.010 ^{dAB}	2.21 ± 0.04 ^{aA}	2.35 ± 0.03 ^{aA}	30.14 ± 0.42 ^{bA}	9.88 ± 0.79 ^{aA}	−4.26 ± 1.14 ^{bA}	−23.4 ± 7.2 ^{bA}	8.6 ± 4.9 ^{bA}	3.04 ± 0.80 ^{aA}
	CP5	0.283 ± 0.010 ^{cA}	2.23 ± 0.01 ^{aA}	2.38 ± 0.01 ^{aA}	31.91 ± 0.45 ^{abA}	11.82 ± 0.29 ^{aA}	−2.69 ± 0.57 ^{bA}	−12.8 ± 2.9 ^{bA}	16.4 ± 2.0 ^{abA}	3.33 ± 0.08 ^{aB}
	IUS	0.311 ± 0.008 ^{bA}	2.03 ± 0.06 ^{bA}	2.18 ± 0.06 ^{bA}	32.58 ± 0.16 ^{aA}	11.71 ± 0.31 ^{aB}	−2.53 ± 0.29 ^{bB}	−12.2 ± 1.2 ^{bB}	16.4 ± 0.8 ^{abB}	3.77 ± 0.17 ^{aB}
70	Control	0.346 ± 0.013 ^{aB}	2.07 ± 0.12 ^{bcA}	2.27 ± 0.13 ^{abA}	25.15 ± 0.26 ^{bB}	14.75 ± 0.62 ^{aA}	0.51 ± 1.00 ^{aA}	1.9 ± 3.9 ^{aA}	40.0 ± 6.0 ^{aA}	7.21 ± 0.59 ^{aA}
	CP2.5	0.231 ± 0.009 ^{cB}	2.18 ± 0.04 ^{abA}	2.31 ± 0.04 ^{abA}	31.67 ± 0.49 ^{aA}	10.67 ± 0.07 ^{bcA}	−3.76 ± 0.36 ^{cA}	−19.4 ± 1.7 ^{cA}	11.2 ± 0.9 ^{bA}	3.92 ± 0.11 ^{bA}
	CP5	0.254 ± 0.005 ^{cB}	2.26 ± 0.02 ^{aA}	2.41 ± 0.02 ^{aA}	28.91 ± 1.57 ^{aAB}	11.36 ± 1.13 ^{bA}	−1.69 ± 0.78 ^{bA}	−8.7 ± 4.8 ^{bA}	20.5 ± 5.8 ^{bA}	3.65 ± 0.02 ^{bA}
	IUS	0.285 ± 0.007 ^{bB}	2.01 ± 0.03 ^{cA}	2.16 ± 0.04 ^{bA}	30.51 ± 1.68 ^{aA}	9.16 ± 0.53 ^{cC}	−2.58 ± 0.28 ^{bcB}	−15.7 ± 1.8 ^{bcB}	12.1 ± 1.7 ^{bC}	3.01 ± 0.36 ^{bC}

Values are mean ± SD (n = 3). Different lowercase letters within a drying temperature indicate significant differences among pretreatments, and different uppercase letters within a pretreatment indicate significant differences among drying temperatures (Tukey's HSD, $p < 0.05$). a_w, water activity of the powder at 25 °C; RR, rehydration ratio (mass of the rehydrated sample/mass of the dried sample); RR_{DM}, rehydration ratio per unit dry matter (mass of the rehydrated sample/dry-matter mass of the dried sample); h°, hue angle expressed as a signed angle relative to the +a* axis; BI (CIELAB), browning index calculated from L*, a* and b*; BI (A420), absorbance-based browning index, expressed as absorbance units per g of powder as analysed, not corrected for the moisture content of the powder.

The rehydration ratio showed no significant temperature effect within any group ($p \geq 0.51$) and no significant pretreatment × temperature interaction ($p = 0.78$). The plasma groups rehydrated 5–11% more than the control ($p < 0.05$, except CP2.5 at 70 °C), whereas IUS did not differ significantly from it. Part of that difference follows from residual moisture, since RR is expressed per unit dried mass and the plasma groups ended drying about three percentage points lower in moisture content; per unit dry matter (RR_{DM}, Table 3) the difference narrowed and was significant in only three of the six plasma–control comparisons. The shorter characteristic drying times after IUS therefore did not coincide with a higher rehydration ratio. Higher rehydration after plasma pretreatment has also been reported for whole wolfberry, by 7–16% [46], and for whole blueberries rehydrated at 25 °C for 24 h, as here [20].

The pretreatment × temperature interaction was significant for every colour variable ($p \leq 0.0004$), so colour is described within temperatures. The powders were dark purple, and a* was numerically lower in every pretreated group than in the control, significantly so in five of the nine comparisons. CP2.5 was more negative in b* than the control at all three temperatures, whereas the direction of the b* response in CP5 and IUS depended on temperature. Lightness differed only at 60 and 70 °C (Table 3).

The CIELAB browning index was 64–72% lower in CP2.5 than in the control at all three temperatures ($p < 0.05$), with CP5 numerically intermediate. Both plasma groups were also below the control in BI(A420) at 50 and 70 °C, by 16–49%, without differing from each other, and no treatment effect was detected at 60 °C ($p = 0.18$). Wherever a treatment effect was detected, both indices placed the plasma groups below the control, but they did not rank the two doses identically, consistent with their different measurement bases. IUS behaved differently, its BI(A420) being the highest of all samples at 50 °C and among the lowest at 70 °C. In the control, both indices rose between 50 and 70 °C ($p \leq 0.021$), whereas neither changed with temperature in CP2.5.

Because the drying duration was equal within a temperature, the lower indices in the plasma groups did not result from a shorter residence time in the oven. The product temperature history was not recorded, however, and the plasma groups, which dried faster, may have spent a longer part of the run at a lower moisture content; the contribution of this difference to browning cannot be separated from that of the pretreatment itself. A lower CIELAB browning index has also been reported after plasma pretreatment of hot-air-dried apple slices, where the pretreatment also shortened drying [47]. In dried chokeberry, colour changes have been associated with the anthocyanin content [7].

3.6. Bioactive Compounds and Antioxidant Capacity

The phenolic, flavonoid and anthocyanin contents and the two antioxidant assays are given in Table 4 with the freeze-dried reference. In the two-way analysis, pretreatment affected TPC, TFC, TMA and CUPRAC ($p \leq 0.002$) but not DPPH inhibition ($p = 0.073$), and no pretreatment × temperature interaction was detected for any of the five responses ($p \geq 0.14$). CP2.5 gave the highest mean TPC, TFC, TMA and CUPRAC at every temperature, followed numerically by CP5, but the two plasma treatments never differed significantly and IUS never differed from the control. TFC gave the most consistent response, being 10–20% higher in CP2.5 than in the control at all three temperatures ($p < 0.05$). TPC did not differ from the control at any temperature, whereas TMA and CUPRAC did so only at 70 °C, where CP5 also exceeded the control in TMA. DPPH inhibition ranged from 73 to 84%, with no significant differences among pretreatments at the dilution used. Both antioxidant assays are in vitro chemical measurements and do not indicate antioxidant activity in a biological system.

Table 4. Total phenolic (TPC), total flavonoid (TFC) and total monomeric anthocyanin (TMA) contents, DPPH radical-scavenging activity and cupric ion reducing antioxidant capacity (CUPRAC) of black chokeberry dried at 50, 60 and 70 °C after cold plasma (CP2.5, CP5) and indirect ultrasound (IUS) pretreatments, and of the freeze-dried reference (FD).

T (°C)	Pretreatment	TPC (mg GAE/100 g DM)	TFC (mg CE/100 g DM)	TMA (mg C3G/100 g DM)	DPPH inhibition (%)	CUPRAC (mg TE/g DM)
50	Control	2172 ± 103 ^{abA*}	435 ± 14 ^{bA*}	455.9 ± 13.9 ^{abA*}	78.3 ± 7.0 ^{aA}	262.5 ± 9.4 ^{abA*}
	CP2.5	2380 ± 86 ^{aA*}	521 ± 23 ^{aA*}	486.0 ± 17.0 ^{aA*}	81.9 ± 4.9 ^{aA}	284.1 ± 12.6 ^{aB*}
	CP5	2226 ± 32 ^{abA*}	486 ± 10 ^{abA*}	467.7 ± 15.7 ^{abAB*}	80.4 ± 3.7 ^{aA}	270.6 ± 6.4 ^{abB*}
	IUS	2116 ± 137 ^{bA*}	452 ± 31 ^{bA*}	442.2 ± 13.5 ^{bA*}	80.9 ± 3.1 ^{aA}	256.2 ± 6.2 ^{bB*}
60	Control	2273 ± 85 ^{aA*}	450 ± 17 ^{bA*}	476.2 ± 18.0 ^{aA*}	80.4 ± 3.8 ^{aA}	281.9 ± 12.7 ^{aA*}
	CP2.5	2464 ± 87 ^{aA*}	497 ± 13 ^{aA*}	495.1 ± 15.2 ^{aA*}	83.4 ± 2.9 ^{aA}	299.2 ± 8.1 ^{aAB*}
	CP5	2412 ± 185 ^{aA*}	474 ± 17 ^{abA*}	489.3 ± 12.5 ^{aA*}	83.8 ± 3.1 ^{aA}	296.1 ± 15.9 ^{aAB*}
	IUS	2347 ± 116 ^{aA*}	448 ± 12 ^{bA*}	469.2 ± 20.2 ^{aA*}	82.6 ± 2.9 ^{aA}	283.4 ± 7.7 ^{aA*}
70	Control	2297 ± 107 ^{aA*}	416 ± 11 ^{bcA*}	385.4 ± 17.1 ^{cB*}	73.1 ± 2.6 ^{aA}	288.5 ± 10.5 ^{bA*}
	CP2.5	2490 ± 50 ^{aA*}	498 ± 26 ^{aA*}	440.1 ± 18.5 ^{aB*}	78.1 ± 2.8 ^{aA}	318.7 ± 13.4 ^{aA*}
	CP5	2404 ± 49 ^{aA*}	462 ± 29 ^{abA*}	431.9 ± 17.5 ^{abB*}	81.0 ± 4.9 ^{aA}	299.4 ± 7.6 ^{abA*}
	IUS	2338 ± 92 ^{aA*}	390 ± 12 ^{cB*}	390.2 ± 10.8 ^{bcB*}	72.6 ± 2.3 ^{aB}	278.8 ± 7.5 ^{bA*}
FD	–	2786 ± 58	663 ± 19	586.5 ± 18.4	–	345.2 ± 4.2

Values are mean ± SD (n = 3). TPC, TFC, TMA and CUPRAC are expressed on a dry-matter (DM) basis using the moisture content of each powder; DPPH is given as percentage inhibition at the extract preparation and dilution stated in Sections 2.7 and 2.9, is not expressed on a DM basis and is not standardised to an equal dry-matter dose. Different lowercase letters within a drying temperature indicate significant differences among pretreatments, and different uppercase letters within a pretreatment indicate significant differences among drying temperatures (Tukey's HSD, $p < 0.05$). * Significantly different from the freeze-dried reference (Dunnett's test, $p < 0.05$). DPPH inhibition of the freeze-dried reference was measured at a different dilution and is therefore not included in the comparison.

Temperature affected all five responses, but in different directions. TMA had its lowest mean at 70 °C in every group (within-pretreatment $p \leq 0.015$), 15.5% below the 50 °C value in the control and 7.7–11.8% below it in the pretreated groups; 50 and 60 °C never differed. CUPRAC moved in the opposite direction, rising 9–12% between 50 and 70 °C, with significant temperature effects in the three pretreated groups ($p = 0.008$–0.033); its highest mean was at 70 °C in the control and the plasma groups but at 60 °C in IUS. DPPH inhibition also varied with temperature ($p = 0.002$) but did not follow CUPRAC, its lowest group mean occurring at 70 °C. Temperature also affected TPC in the two-way analysis ($p = 0.001$), although no within-pretreatment contrast reached significance. Because drying lasted 35.5, 23.5 and 14.5 h at 50, 60 and 70 °C, temperature and duration were not varied independently, and these comparisons carry both. The same divergence between the two assays has been reported for hot-air-dried chokeberry, in which the cupric ion reducing capacity exceeded that of the fresh fruit while DPPH and FRAP values fell [48]. Greater losses of anthocyanins than of total polyphenols during hot-air drying have also been reported for chokeberry [49].

Every hot-air-dried sample contained less TPC, TFC, TMA and CUPRAC than the freeze-dried reference (Table 4). The hot-air-dried samples reached 76–89% of the reference for TPC, 59–79% for TFC, 66–84% for TMA and 74–92% for CUPRAC, highest in CP2.5 and lowest in the control or IUS. DPPH inhibition of the reference was measured at a two-fold dilution and is not compared. Fresh fruit was not analysed, so these values describe levels relative to the freeze-dried reference rather than retention relative to fresh fruit. Freeze-drying also gave the highest phenolic content and antioxidant values among the drying methods compared for whole chokeberry [4].

The higher mean values in the plasma groups coincided with their faster moisture removal, but the absence of a significant difference between CP2.5 and CP5 does not establish an exposure-dependent compositional effect. A higher TPC after plasma pretreatment has also been reported for whole blueberries [19]. Despite its shorter drying times, IUS did not differ from the control in any of the five responses. Whether the plasma groups retained more of these compounds because the fruit spent less time at high moisture content, or because the treatment altered the tissue before drying, cannot be decided from these data; no measurement was made of enzyme activity or of any stress response of the fruit to the treatment, and no such mechanism is proposed here.

3.7. Surface Microstructure

Representative micrographs of the fruit surface after drying at 60 °C are shown in Figure 4, with one representative field presented for each group. The untreated field (Figure 4A) showed a finely granular, largely continuous surface with broad folds. The CP2.5 field (Figure 4B) was ridged and contained a fissure-like feature, whereas the CP5 field (Figure 4C) showed pronounced creasing, local surface openings and a depressed region. The IUS field (Figure 4D) was pitted and rough but largely continuous. In apple slices, plasma pretreatment has been associated mainly with surface etching and ultrasound with disruption of the internal tissue [11]; whether the same distinction applies to a whole fruit with an intact cuticle was not tested here, since the interior was not imaged and no direct-sonication group was included. These fields illustrate surface topography after drying but do not quantify the frequency or severity of the observed features among treatments, and the micrographs were interpreted qualitatively rather than as evidence of treatment ranking or moisture-transport mechanisms.

3.8. Limitations of the Study

Several features of the design limit the interpretation of these results. Drying duration was fixed within each temperature, so the groups ended drying at different moisture contents, and the compositional, colour and rehydration comparisons describe fruit dried for the same time rather than to the same end point; temperature and duration were likewise not varied independently. Expression per unit dry matter corrects the denominator for residual moisture but does not equalise the moisture and temperature histories of the groups, so the compositional differences describe the outcome of the protocols rather than a pretreatment effect at a common end point. Fresh-fruit bioactive compound contents were not measured, so comparisons with the freeze-dried reference do not quantify retention relative to fresh fruit. Plasma was examined at two exposure times under one device setting and ultrasound at one frequency, duration, loading and operating mode, so neither treatment was optimised. The study cannot locate an optimum, and the effects of other exposure times or durations, of other ultrasound frequencies such as 20 kHz, or of programmed pulsed sonication cannot be inferred from the present results. The plasma treatment was characterised by exposure time, nozzle distance, air flow rate and fruit surface temperature; the power dissipated in the discharge, the energy absorbed by the fruit, the humidity and temperature of the working gas and the reactive species formed were not measured, and the acoustic power delivered to the fruit was likewise not measured. Neither an immersion-only control nor a direct-sonication group was included, so the IUS response combines the effects of sonication and of 30 min of water immersion, and the two sonication configurations cannot be compared. Since probe and bath sonication have produced different structural effects in the same tissue [28], the results do not indicate how the treatment would perform under probe or direct-bath sonication, at larger loadings or at larger processing volumes; scale-up would require validation of acoustic-field uniformity, loading, vessel geometry and temperature

control. Energy consumption was not measured, and the treatments were not matched for energy input, so the shorter characteristic drying times cannot be converted into an energy saving.

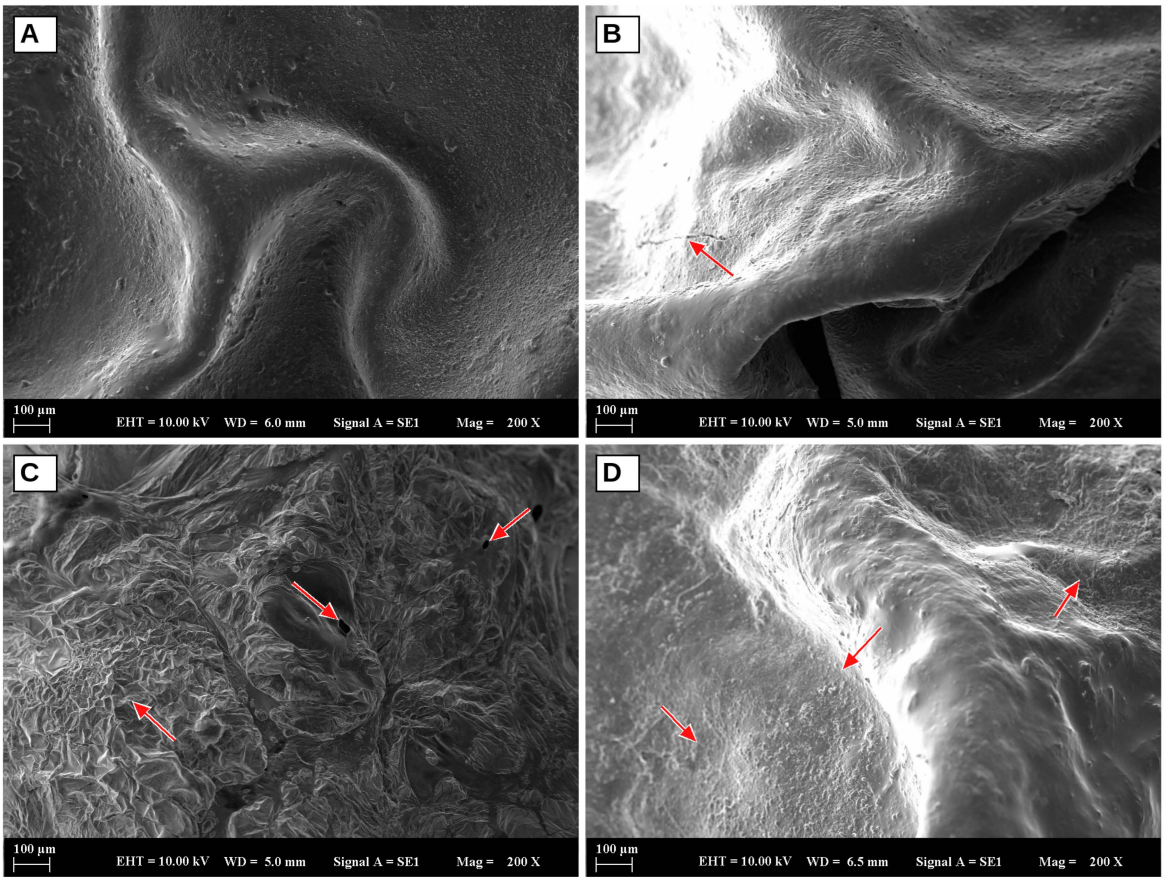


Figure 4. SEM micrographs (200×) of the surface of whole black chokeberry fruits dried at 60 °C: (**A**) Control, (**B**) CP2.5, (**C**) CP5 and (**D**) IUS. Arrows indicate a fissure-like feature (**B**), surface openings and pronounced wrinkling (**C**) and surface pitting and roughness (**D**). Scale bars, 100 µm. One representative field is shown per group; the micrographs are illustrative and were not used for quantitative morphometric inference.

The product temperature during drying, the course of shrinkage, enzyme activities and any stress response of the fruit to the treatments were not measured, so the mechanisms behind the kinetic and compositional differences remain untested. D_{eff} was estimated with a fixed initial volume-equivalent radius without shrinkage correction and is interpreted as an apparent comparative coefficient, and the activation energies rest on three temperature levels each. At 50 °C, eight of the twelve t_{10} estimates depended on interpolation across an overnight weighing interval, and the model-based comparison in Section 3.1 was a sensitivity check rather than an independent validation. The micrographs were qualitative, were obtained on dried fruit at one temperature and do not separate the effect of pretreatment from that of drying. Relative humidity in the drying chamber was neither controlled nor monitored. The fruit came from a single harvest lot of one cultivar. The experiments were carried out at laboratory scale with 30 g batches, so the kinetic parameters and the comparisons are specific to this protocol.

4. Conclusions

Cold plasma and indirect ultrasound both accelerated moisture removal during the hot-air drying of whole black chokeberry at 50, 60 and 70 °C, but their effects on the dried fruit differed. CP2.5 gave the strongest kinetic response, reducing t_{10} by 19–25% and increasing the apparent D_{eff} by 24–34%. It also gave the highest mean TPC, TFC, TMA and CUPRAC values among the hot-air-dried groups, with TFC significantly above the control at every temperature, together with a CIELAB browning index 64–72% below the control. Doubling the plasma exposure reduced the kinetic benefit; the two plasma groups did not differ significantly in any compositional variable, so a compositional advantage of CP2.5 over CP5 was not demonstrated, although CP5 produced the lowest shrinkage. IUS reduced t_{10} by 11–13% but showed no significant compositional or antioxidant advantage over the control, and its effect cannot be separated from that of the accompanying water immersion.

Drying temperature had a larger effect on drying time than any pretreatment: increasing it from 50 to 70 °C reduced t_{10} of untreated fruit by 59%, while TMA means were 8–15% lower at 70 °C than at 50 °C. Because temperature and drying duration were not varied independently, this compositional difference cannot be attributed to temperature alone. Apparent activation energies were numerically lower after pretreatment, but the Arrhenius slopes did not differ significantly. These conclusions apply to the specific treatment settings, drying protocol and fruit lot used; the treatments were not optimised or matched for energy input, and the mechanisms by which they altered drying were not examined. Dose series for both pretreatments, drying to a common end point and measurement of product temperature and energy use would be needed to extend them.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/pr14233830/s1, Table S1, Constants and goodness-of-fit statistics of the four thin-layer models fitted to the drying curves of whole black chokeberry fruits dried at 50, 60 and 70 °C after cold plasma (CP2.5, CP5) and indirect ultrasound (IUS) pretreatments; Table S2, Sensitivity of the apparent effective moisture diffusivity to the moisture-ratio window used for the linear regression of ln(MR) on time, for whole black chokeberry fruits dried at 50, 60 and 70 °C after cold plasma (CP2.5, CP5) and indirect ultrasound (IUS) pretreatments.

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