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Research Article

Effects of Different Drying Conditions on Key Quality Parameters of Pink Peppercorns (*Schinus terebinthifolius* Raddi)

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Pink peppercorns are among the most sophisticated condiments in the international cuisine. This culinary spice is obtained from dried fruits of *Schinus terebinthifolius* Raddi, a species native to South America. In this work, a methodology for the assessment of pink peppercorn quality under various drying conditions was defined. Experiments were performed in a pilot tray dryer, which ensured integrity of the product. A central composite rotatable design with 11 experiments was devised to study the influence of drying air temperature (35–75°C) and air velocity (0.3–0.9 m/s) on product quality, assessed by moisture content, color (CIELAB system), and volatile compounds. The essential oils of fresh and dried fruits were extracted by hydrodistillation and analyzed by gas chromatography coupled to mass spectrometry. Air temperature had the greatest influence on the quality parameters under study, while air velocity had no statistically significant effect. Considering all quality criteria, temperatures between 40 and 55°C provided the best compromise, yielding an adequate moisture content in the dried product without dramatic degradation of color and essential oil.

1. Introduction

Drying is still the most popular method for preservation of agricultural products (e.g., fruits, vegetables, herbs, and spices), ensuring microbial safety of various biological materials. Nonetheless, this method has several disadvantages and limitations. For example, contact between the dried material and hot air causes degradation of key flavor compounds and nutritional substances, and there is a risk of volatilization of volatile compounds during the drying operation. In addition, undesirable changes in product color, possibly due to Maillard reactions, can be observed in the processed material [1–3]. Both locally and internationally, numerous researchers have attempted to study the influence of drying on the quality of various products, corroborating the importance of

additional studies in this area [2–8]. These reports include those on Australian nectarines [4], lemon myrtle leaves (*Backhousia citriodora*) [5], oregano (*Origanum vulgare*) [2], lemon verbena (*Lippia citriodora*) [6], curry leaves (*Murraya koenigii* L. Spreng.) [7], and lemon peels (*Citrus limon* cv. *lunari*) [8].

Pink peppercorns, also called pink pepper, are among the most sophisticated condiments in the international cuisine, adding a slightly spicy and sweet taste to food. In recent years, their consumption has shown significant growth in the international market. This culinary spice is obtained from ripe fruits of *Schinus terebinthifolius* Raddi, a species native to South America. In the food industry, these fruits are usually sold in dried form and should have a bright red color and a spherical shape in the absence of broken shells.

It should be noted that *S. terebinthifolius* is not related to the pepper family (Piperaceae); rather, this plant belongs to the Anacardiaceae family, being a relative of cashew and mango [9]. Besides its use as a condiment, *S. terebinthifolius* has great commercial potential owing to its medicinal, cosmetic, and pharmaceutical properties [10–12].

In Brazil, production of pink peppercorns is carried out empirically, and the drying step is a crucial operation. *S. terebinthifolius* fruits are dried to inhibit microbial growth and quality decay, thus adding value to the fruits. Another benefit of drying is reduced weight; the removal of large amounts of water from the material makes transport more economical. Nonetheless, if drying is not performed properly, the quality and marketability of the pink peppercorn are affected. In the production of pink peppercorns, appearance (color and integrity) and flavor are among the most important quality criteria. Thus far, few studies have addressed the drying of *S. terebinthifolius* fruits. Silva et al. [13] studied the drying process of *S. terebinthifolius* fruits with an emphasis on the development of models for drying kinetics. On the other hand, no scientific work has yet been reported regarding the effects of different drying conditions on pink peppercorn quality (integrity, color, and volatiles). Studies on this process are necessary for improving pink peppercorn quality and would allow for a better understanding of the phenomena involved, as well as the behavior of the material during the drying operation. Therefore, the main aim of this work was to study the effect of different drying conditions on key quality parameters of pink peppercorns, including physical and physicochemical parameters and volatile compounds.

2. Materials and Methods

2.1. Raw Material. Ripe fruits of *S. terebinthifolius* were manually collected from native trees in Aracruz (Espírito Santo, Brazil) in May 2015. A voucher specimen (number 188116) was deposited at the Herbarium of the Institute of Biology, University of Campinas, Brazil. Fresh fruits were manually separated from impurities (e.g., unripe fruits, leaves, twigs, and stones) and stored in closed plastic bags at -18°C until use.

2.2. Morphological and Physicochemical Parameters. Knowledge of the physical properties of food particles is fundamentally important for the design, modeling, and optimization of many food processing operations such as storage and drying. Although a considerable amount of data on important food properties have already been reported for many agricultural crops, no detailed study on the morphological and physicochemical properties of *S. terebinthifolius* fruits has been performed hitherto. Thus, characterization of *S. terebinthifolius* fruits (fresh and dried) was performed to evaluate moisture content, color (CIELAB system), Sauter mean diameter, density (apparent and bulk), and weight of 1000 fruits. All assays were performed in triplicate. Dry basis moisture content (M) and wet basis moisture content (M^*) were determined by drying the samples in a vacuum oven (Quimis®, Q819V2) for 24 h at 70°C [14]; these conditions led

to constant final weights. The color of *S. terebinthifolius* fruits was determined using a handheld Mini Scan XE colorimeter (Hunter Lab®, model 4510-L, Reston, VA, USA) with a D65 illuminant and a 10° viewing angle. The color was reproduced using the CIELAB color space, where L^* is lightness, a^* is red-green proportion, and b^* is blue-yellow proportion. Calibration of the colorimeter was performed daily using black and white tiles. The total color difference (ΔE) was determined using

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2}. \quad (1)$$

The subscript “0” in (1) refers to the color parameters of pink peppercorns obtained from the local market. The Sauter mean diameter (d_p) was calculated to represent the mean diameter of *S. terebinthifolius* fruits, according to

$$d_p = \frac{1}{\sum_{i=1}^n (x_i/D_i)}, \quad (2)$$

where x_i is the mass fraction of particles with mean diameter D_i ; D_i is the mean diameter of particles retained between one test sieve and the subsequent one. A vibratory sieve shaker (Bertel®, 302 model) and mesh sieves 1/4", 3.5, 4, 5, 6, and 7 (Tyler series, USA) were used. To determine the apparent density of a fruit (ρ_p), *S. terebinthifolius* fruits were regarded as spheres ($\Phi_p = 1$); the spherical volume (V_p) was calculated as a function of the Sauter mean diameter. The mass of a fruit (m_p) was determined from the weight of 1000 fruits (W_{1000}), which was measured using a digital scale (SHIMADZU® AU7220; 0.0001 g accuracy) according to the method described by ISTA [15]. Bulk density (ρ_{bulk}) of *S. terebinthifolius* fruits was determined by weighing the contents of a cylindrical container of known volume. The measurement was conducted using a 2-L graduated cylinder. Porosity (ϵ), or void fraction, was estimated from the apparent and bulk densities, as described by Rapusas et al. [16].

2.3. Drying, Hydrodistillation, and Chromatographic Analyses. In general, the experimental procedure consisted of two stages: firstly *S. terebinthifolius* fruits were dried, and secondly morphological and physicochemical parameters were characterized, and volatile compounds were extracted from the dried product. The drying of *S. terebinthifolius* fruits was carried out in a pilot thin-layer dryer (tray dryer), described by Silva et al. [13]. The dryer receives atmospheric air from a fan, and air flow is adjusted by a manual valve. The air heating system consists of four electrical resistors connected to a temperature control system. A fixed bed thickness of 1 cm was used in the experiments. Relative humidity (RH) was measured continuously throughout the experiments using a thermohygrometer (Ummi®). In each drying assay, drying kinetics and rate (see (3)) were analyzed.

$$\text{DR} = \frac{M_{i-1} - M_i}{t_i - t_{i-1}}, \quad (3)$$

where M_i is dry basis moisture content at any time point (t_i) and M_{i-1} is moisture content prior to M_i . Details of the

experimental methodology for drying have been described previously [13]. A central composite rotatable design (CCRD) with 11 experiments (Table 1) was devised to study the influence of drying air temperature and velocity on product quality in terms of final moisture content, Sauter mean diameter, density (apparent and bulk), porosity, weight of 1000 fruits, and color (CIELAB system). The data were processed in STATISTICA® software. The influence of drying on the essential oil of *S. terebinthifolius* fruits was also evaluated.

Extraction of essential oil was accomplished by hydrodistillation in a Clevenger apparatus. Before each extraction, fruits were ground in a hand mill (BOTINI®, model B03). After that, the crushed fruits and 1 L of distilled water were mixed in a 2-L round-bottom flask. The system was heated (at level 4 power) using a 2-L heating mantle (Fisaton®, model 202, 480 W). After hydrodistillation, the distillate (oil and water) was transferred to a separatory funnel, and the essential oil was extracted three times with anhydrous dichloromethane (Synth®). Two phases were obtained: an aqueous phase and an organic phase containing oil and dichloromethane. The organic phase was collected and dried over anhydrous sodium sulfate (Êxodo Científica®). Dichloromethane was evaporated using a rotatory evaporator (Buchi®, model R-200) under vacuum at 40°C. Next, the mass of essential oil was determined, and the extraction yield on a dry basis (Y) was calculated according to

$$Y = \frac{m_o}{m_d} 100, \quad (4)$$

where m_o is the mass of essential oil, and m_d is the dry mass of the raw material fed into the extractor. Quality attributes were determined and compared with those of fresh fruits. For example, the yield ratio (YR) was calculated using

$$\text{YR} = \frac{Y}{Y_f}, \quad (5)$$

where Y and Y_f are the extraction yields of the essential oil extracted from dried and fresh fruits of *S. terebinthifolius*, respectively.

Quantitative and qualitative analyses of the essential oil were carried out using gas chromatography coupled to mass spectrometry (GC-MS). The samples were analyzed on a gas chromatograph (Agilent Technologies, HP-6890, USA) equipped with a mass spectrometer (Agilent Technologies, HP5975, USA) operating in electron impact mode (70 eV). A HP-5MS® capillary column (30 m × 250 μm, 0.25 μm phase thickness) was used. The carrier gas was helium, and the flow rate was 1 mL/min. Injector and detector temperatures were 220 and 250°C, respectively. Oven temperature was programmed with a ramp from 60 to 240°C at a rate of 3°C/min. Prior to GC-MS analysis, samples were solubilized in ethyl acetate (10 mg/mL). The injection volume was 1 μL; injection was performed in split mode at a ratio of 1:30. Individual constituents were identified by comparison of their linear retention index (RI) [17] and mass spectra with published data [18]. The retention index was calculated using a homologous series of *n*-alkanes, C₈–C₂₂. The relative percentages obtained from chromatogram peak areas served as indicators of the amounts of volatile components [19].

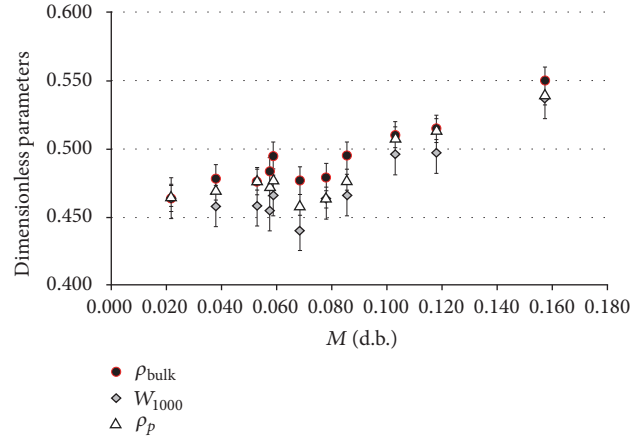


FIGURE 1: Dimensionless physical parameters (relative to fresh fruits) as a function of moisture content of *S. terebinthifolius* fruits. M : moisture content on dry basis; ρ_{bulk} : bulk density; W_{1000} : weight of 1000 fruits; ρ_p : apparent density.

3. Results and Discussion

In this work, the quality of pink peppercorns under different drying conditions was evaluated. Experiments were designed (Table 1) to study the influence of drying air temperature (35–75°C) and air velocity (0.3–0.9 m/s) on product quality, measured primarily by means of moisture content, color, and volatile compounds. Tables 2–4 show the results for all parameters analyzed in this study. Considering all quality criteria, drying air temperature had the greatest influence on the quality parameters of pink peppercorns under study; drying air velocity had no significant effects. These results suggest that resistance to moisture movement at the surface of the fruits was negligible compared with internal resistance [20]. Accordingly, the effects of drying air temperature are discussed in detail hereafter.

3.1. Effects on Moisture Content. Appropriate moisture content in pink peppercorns is necessary to avoid microbial growth and product degradation. Therefore, low moisture contents are recommended ($M^* < 12\%$) [21]. As shown in Table 2, the final moisture content on a wet basis ranged from 2.1% (75°C and 0.6 m/s) to 13.6% (35°C and 0.6 m/s). Thus, a drying air temperature of 35°C resulted in a moisture content above 12%. Furthermore, drying at lower temperatures required longer operation time. As shown in Table 1, the equilibrium drying time (t_e) ranged from 4 h (75°C and 0.6 m/s) to 14 h (35°C and 0.6 m/s). Thus, the increase in temperature from 35 to 75°C, and the consequent decrease in relative humidity (from 31% to 5%), increased the drying rate and reduced the drying time and moisture content of *S. terebinthifolius* fruits. As shown in Table 2, an increase in temperature also resulted in a decrease in density (apparent and bulk) and in the weight of 1000 fruits, as expected. These changes were due to changes in fruit moisture content. This phenomenon can be seen in Figure 1, which depicts the nonlinear dependence of these physical parameters on the moisture content of *S. terebinthifolius* fruits.

TABLE 1: Operating conditions for drying of *S. terebinthifolius* fruits.

Assay	T ($^{\circ}\text{C}$) ^a	V (m/s) ^a	RH (%)	M_o (d.b.)	M_o^* (%)	t_e (min)
1	40.9 (-1)	0.39 (-1)	22	1.62	61.8	690
2	69.1 (+1)	0.39 (-1)	6	1.62	61.8	270
3	40.9 (-1)	0.81 (+1)	23	1.62	61.8	705
4	69.1 (+1)	0.81 (+1)	7	1.63	62.0	300
5	35.0 (-1.41)	0.60 (0)	31	1.62	61.8	840
6	75.0 (+1.41)	0.60 (0)	5	1.64	62.1	240
7	55 (0)	0.30 (-1.41)	11	1.64	62.1	495
8	55 (0)	0.90 (+1.41)	13	1.64	62.1	510
9	55 (0)	0.60 (0)	12	1.63	62.0	495
10	55 (0)	0.60 (0)	13	1.62	61.8	510
11	55 (0)	0.60 (0)	13	1.62	61.8	480

T : drying air temperature; V : air velocity; RH: air relative humidity; M_o : initial moisture content on dry basis of *S. terebinthifolius* fruits; M_o^* : initial moisture content on wet basis; t_e : final drying time; ^acoded values in parentheses.

TABLE 2: Physical parameters of *S. terebinthifolius* fruits (fresh and dried under the operating conditions of Table 1).

Assay	M_e (d.b.)	M_e^* (%)	ρ_{bulk} (g/mL)	W_{1000} (g)	d_p (mm)	ρ_p (g/mL)	ε (—)
1	0.1030	9.34	0.277	27.06	4.92	0.433	0.360
2	0.0378	3.64	0.260	25.00	4.92	0.401	0.351
3	0.1180	10.5	0.280	27.11	4.91	0.438	0.362
4	0.0528	5.01	0.259	25.01	4.90	0.407	0.364
5	0.1570	13.6	0.299	29.30	4.96	0.460	0.350
6	0.0216	2.12	0.252	25.31	4.96	0.397	0.365
7	0.0574	5.43	0.263	24.82	4.90	0.403	0.348
8	0.0778	7.22	0.260	25.30	4.96	0.395	0.342
9	0.0586	5.54	0.269	25.42	4.92	0.407	0.339
10	0.0684	6.40	0.259	24.04	4.90	0.391	0.337
11	0.0854	7.87	0.269	25.43	4.92	0.407	0.339
Fresh fruits	1.62	61.8	0.543	54.57	4.96	0.852	0.358

M_e : equilibrium moisture content on dry basis, determined at the final stage of drying; M_e^* : equilibrium moisture content on wet basis; ρ_{bulk} : bulk density; W_{1000} : weight of 1000 fruits; d_p : Sauter mean diameter; ρ_p : fruit apparent density; ε : porosity.

TABLE 3: Color parameters (CIELAB system) of *S. terebinthifolius* fruits (dried under the operating conditions of Table 1).

Assay	L^*	a^*	b^*	ΔE
1	25.72	34.17	22.45	3.16
2	23.90	26.97	16.42	6.91
3	26.52	32.65	21.62	1.41
4	24.70	27.95	18.04	5.01
5	26.41	32.19	21.03	0.70
6	21.12	26.61	17.43	8.19
7	26.50	30.91	19.00	1.74
8	26.49	30.90	19.04	1.72
9	25.83	32.01	20.24	0.77
10	26.16	31.53	19.89	0.78
11	25.23	30.77	18.41	2.68
Commercial pink peppercorn	26.59	31.94	20.40	—

L^* : darkness/whiteness; a^* : greenness/redness; b^* : blueness/yellowness; ΔE : total color difference.

The drying kinetics for all operational conditions listed in Table 1 are presented in Figures 2 and 3. As noted, drying kinetics were not a linear function of time, and moisture content decreased exponentially with drying time; the last step was represented by an almost horizontal line, which marked the end of the drying process (equilibrium moisture content). As shown in Figures 2 and 3, the effects of air velocity on drying kinetics and rate were small compared with the large effects of air temperature. The increase in drying rate at higher temperatures is related to increased moisture diffusivity at higher temperatures. Accordingly, the drying kinetics of *S. terebinthifolius* fruits showed only the falling-rate period of drying. At this stage, mass transfer was limited by diffusion as described by Fick's second law of diffusion. These results are due to the fact that, in biological materials, water molecules tend to be tightly held by the materials [22]. This behavior is consistent with previously published literature on the drying of *S. terebinthifolius* fruits [13] and other fruits and seeds [23–25].

TABLE 4: Chemical composition (GC-MS data) and the yield of essential oil from *S. terebinthifolius* fruits (fresh and dried under the operating conditions of Table 1).

Compound	RI _{lit.} ^a	RI _{cal.} ^b	Relative area (%) ^d											Fresh fruits
			1	2	3	4	5	6	7	8	9	10	11	
α -Pinene	932	933	3.65	3.52	5.12	2.84	4.87	1.49	5.15	3.61	3.72	5.06	2.86	5.36
Sabinene	969	973	2.24	2.14	1.89	1.78	2.20	1.28	2.32	2.03	1.52	2.12	1.50	2.45
β -Myrcene	988	991	12.14	10.90	11.61	11.03	12.16	8.12	11.03	11.73	11.21	11.66	10.91	12.75
α -Phellandrene	1002	1006	18.48	17.84	16.35	16.59	19.64	14.26	19.31	18.69	17.34	17.61	17.16	20.46
δ -3-Carene	1008	1012	18.41	16.81	18.37	16.08	18.60	13.67	17.54	17.00	17.43	17.82	17.04	19.14
n.i. ^c	—	1017	—	—	—	—	—	—	—	—	—	—	0.39	—
<i>p</i> -Cymene	1020	1025	1.36	0.68	2.04	1.21	0.90	0.69	0.66	0.83	1.26	1.09	1.07	0.73
Limonene	1024	1029	17.77	16.30	17.42	16.49	17.91	15.19	16.75	17.43	17.12	15.90	17.37	18.28
n.i. ^c	—	1058	—	—	—	—	—	—	—	—	0.53	—	0.54	—
Terpinolene	1086	1089	1.38	1.26	1.19	1.29	1.33	1.35	1.24	1.33	1.32	1.20	1.44	1.31
n.i. ^c	—	1178	0.68	—	0.74	0.61	0.74	0.58	0.52	0.62	1.00	0.64	1.04	0.68
n.i. ^c	—	1337	—	—	—	—	—	0.61	—	—	—	—	—	—
Caryophyllene	1417	1418	4.50	4.72	4.27	5.20	3.99	5.83	4.08	4.71	4.66	4.52	5.42	3.73
Germacrene D	1484	1480	8.04	8.91	7.13	9.19	6.83	10.90	7.32	8.34	6.47	7.91	8.17	6.25
n.i. ^c	—	1495	—	—	—	—	—	0.71	—	—	—	—	—	—
δ -Cadinene	1522	1523	0.93	1.05	1.10	1.33	1.01	1.45	1.03	1.19	1.65	1.34	1.78	0.94
Elemol	1548	1550	1.38	1.83	1.02	1.82	1.05	6.52	3.50	1.58	1.37	1.55	2.67	0.90
Germacrene B	1559	1555	0.64	0.75	—	0.79	0.57	0.99	0.66	0.72	—	0.67	0.76	—
γ -Eudesmol	1630	1631	4.01	5.70	5.83	5.97	3.39	6.07	3.48	4.94	5.77	4.90	4.27	3.29
β -Eudesmol	1650	1650	1.19	1.75	1.54	1.85	0.96	2.23	1.29	1.47	1.74	1.60	1.38	0.90
α -Eudesmol	1652	1653	2.42	3.16	3.28	3.80	2.03	3.15	2.47	2.67	3.62	3.19	2.38	1.96
n.i. ^c	—	1654	—	—	—	—	—	1.27	—	—	—	—	—	—
n.i. ^c	—	1667	—	0.79	—	—	—	—	—	—	—	—	—	—
n.i. ^c	—	1676	0.81	1.24	1.09	1.40	0.98	2.81	1.64	1.12	1.59	1.21	1.83	0.86
n.i. ^c	—	1776	—	0.65	—	0.73	—	0.85	—	—	0.68	—	—	—
Monoterpenes (%) ^d	—	—	76.1	69.5	74.7	67.9	79.2	56.6	74.5	73.3	72.4	73.1	71.4	81.2
Yield (%) ^e	—	—	5.24	4.87	5.48	4.38	5.38	4.66	5.52	5.64	4.67	5.33	4.90	5.56

^aRetention index [18]; ^bcalculated retention index (average value); ^cnot identified; ^drelative percentage obtained from the peak area of the chromatograms; ^eextraction yield according to (4).

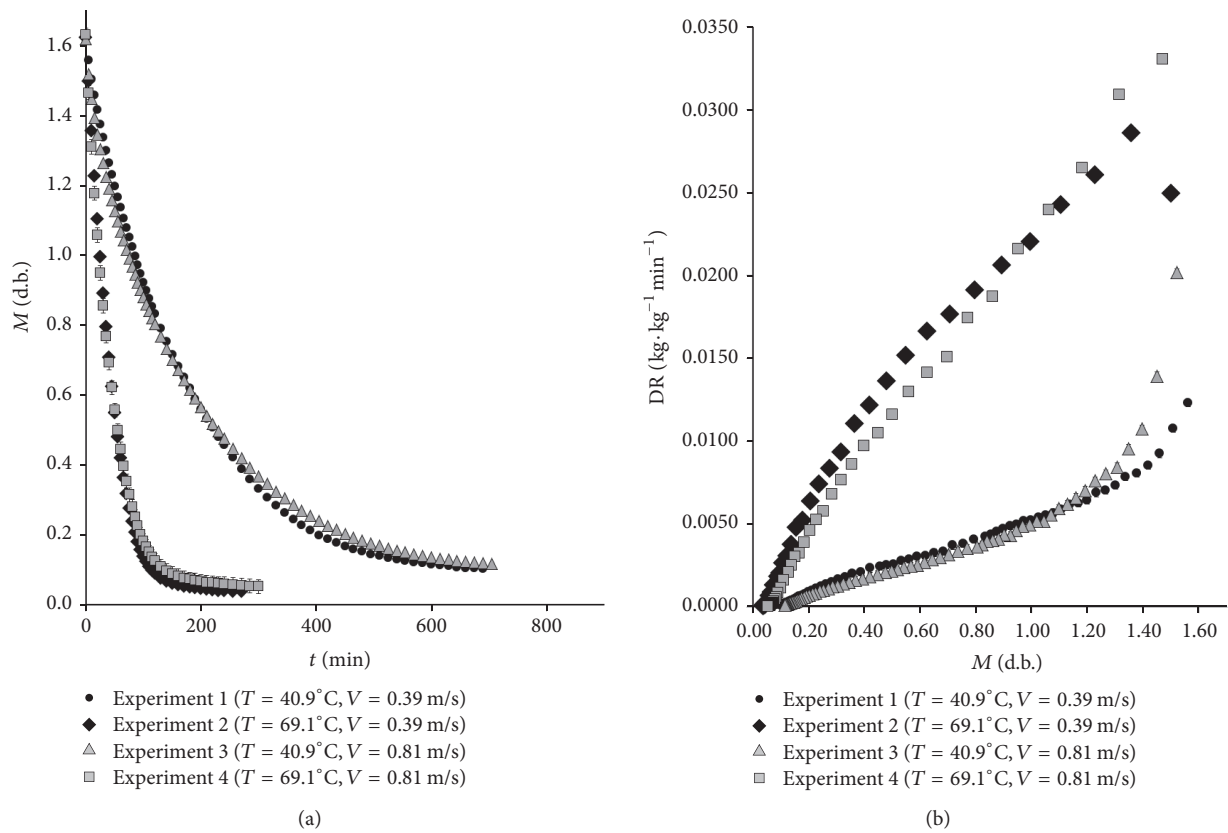


FIGURE 2: (a) Drying kinetics and (b) drying rate for experiments 1–4 (Table 1). M : moisture content on dry basis; t : drying time; T : air temperature; V : air velocity; DR : drying rate.

3.2. Product Integrity. Pink peppercorns should have a bright red color and a spherical shape in the absence of broken shells. Thus, it was possible to evaluate the integrity of *S. terebinthifolius* fruits after drying based on their diameter. As shown in Table 2, the fruits remained intact after the drying process under all conditions investigated, which was possible because of the use of a dryer in a fixed bed (tray dryer). In addition, no shrinkage of the material was observed during drying. Aside from the integrity and moisture content of pink peppercorns, monitoring of parameters such as color and volatile content is necessary to ensure quality of the product. In the next sections of this paper, the effects of drying on these parameters are discussed in detail.

3.3. Effects on Color. The color of pink peppercorns is highly important for commercial acceptability. Pink peppercorns are characterized by a bright red color. This color may be due to phenolic compounds [26] or carotenoids in *S. terebinthifolius* fruits [27]. Nonetheless, specific studies are needed to determine a definitive relationship between these compounds and the color of *S. terebinthifolius* fruits. Table 3 shows the results of the colorimetric analysis (CIELAB system) of *S. terebinthifolius* fruits that were dried under the operating conditions listed in Table 1; as expected for pink peppercorns, the dried fruits had a dark red color (low L^* and positive a^*).

As shown in Table 3, the L^* parameter ranged from 26.52 (40.9°C and 0.81 m/s) to 21.12 (75°C and 0.6 m/s), indicating decreasing luminosity with increasing drying temperature. The a^* parameter ranged from 34.17 (40.9°C and 0.39 m/s) to 26.61 (75°C and 0.6 m/s), indicating decreasing redness with increasing drying temperature. The b^* parameter ranged from 22.45 (40.9°C and 0.39 m/s) to 16.42 (69.1°C and 0.39 m/s), indicating decreasing yellowness with increasing drying temperature. Thus, as shown in Pareto diagrams (Figure 4), increasing the drying air temperature resulted in a statistically significant negative effect on the color parameters, with a confidence level of 95%. Therefore, drying of *S. terebinthifolius* fruits resulted in color degradation, suggesting that higher temperatures cause more pronounced degradation of pigments. These results may be due to either browning reactions or degradation of carotenoid and phenolic compounds at high temperatures. Nevertheless, specific studies are needed to identify a definitive relation between these phenomena and color losses. Unfortunately, information on the nonenzymatic browning of pink peppercorns is still scarce in the literature. Lee et al. [28] suggest that nonenzymatic browning in dried red peppers is due to Maillard reactions. In addition, the loss of red color may have been caused by oxidation of carotenoids in drying air. Klieber [29] states that carotenoids are vulnerable to the

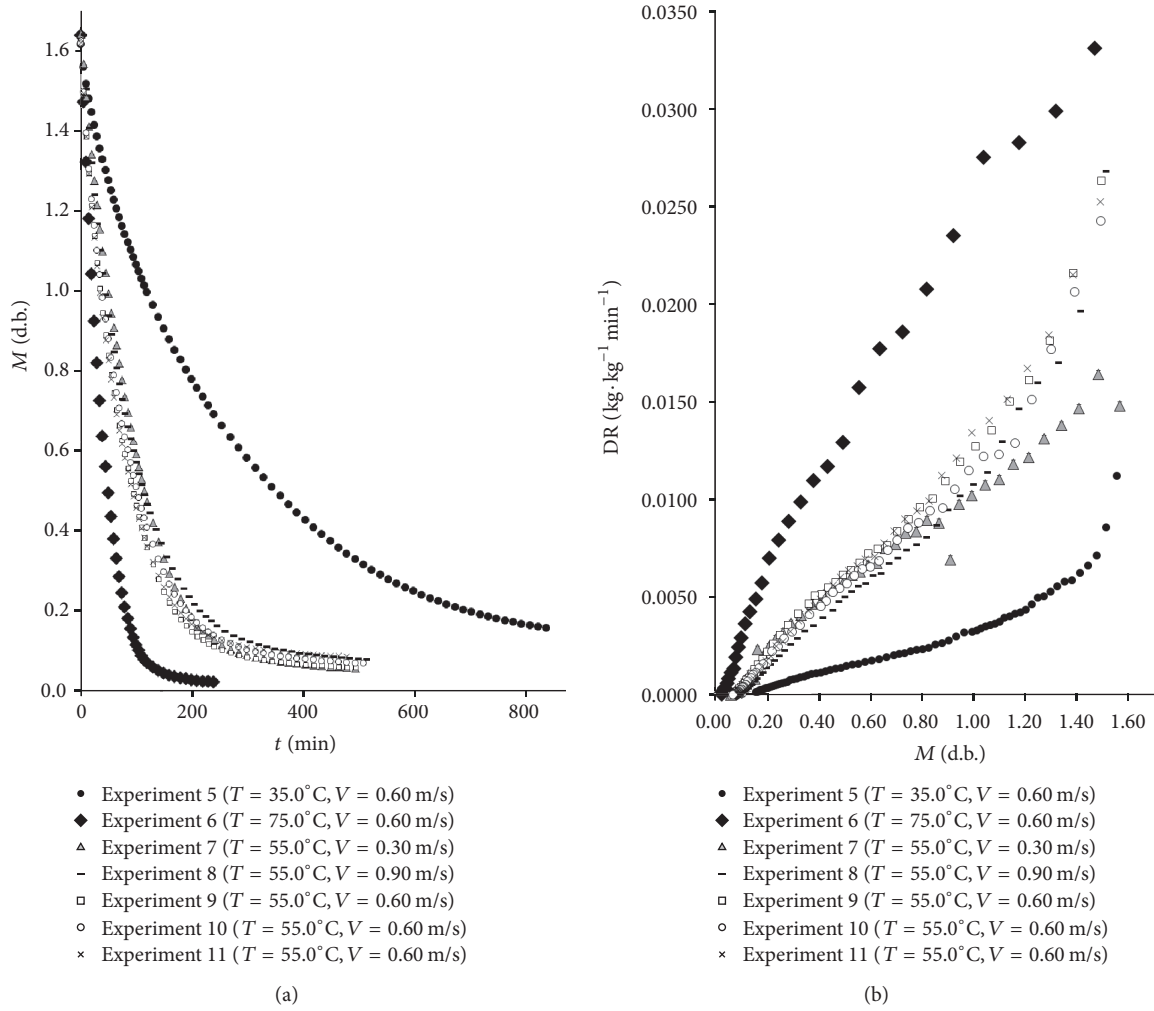


FIGURE 3: (a) Drying kinetics and (b) drying rate for experiments 5–11 (Table 1). M : moisture content on dry basis; t : drying time; T : air temperature; V : air velocity; DR : drying rate.

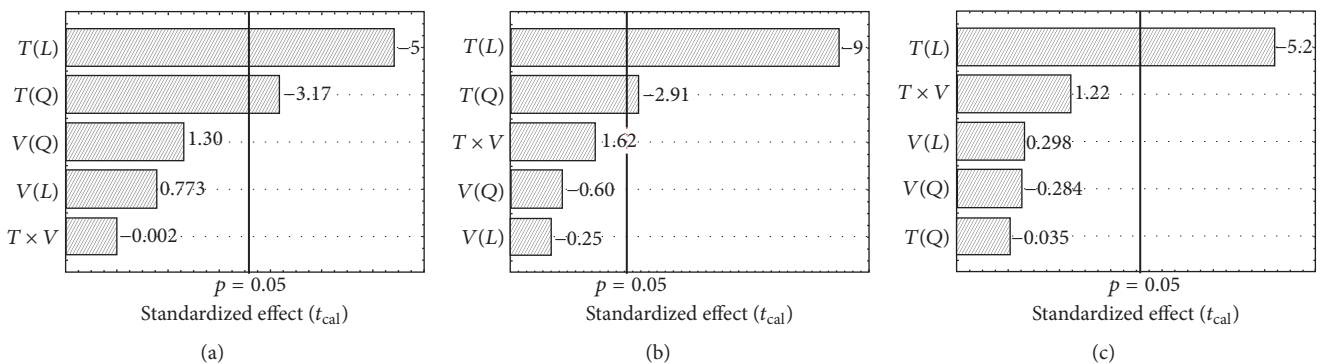


FIGURE 4: Pareto diagrams for color parameters (CIELAB system) of *S. terebinthifolius* fruits: (a) L^* (darkness/whiteness); (b) a^* (greenness/redness); (c) b^* (blueness/yellowness). $T(L)$: linear temperature effect; $T(Q)$: quadratic temperature effect; $V(L)$: linear velocity effect; $V(Q)$: quadratic velocity effect.

effects of heat, light, and high oxygen tension. According to Malchev et al. [30], the degradation rate increases as the drying temperature increases.

Comparison of the data from the pink peppercorns produced in this work with data obtained from commercial

pink peppercorns (Table 3) suggests that low temperatures should be used for drying (35 to 55°C). As shown in Table 3, the total color difference was lower (0.70 to 3.16) for this drying temperature range. In fact, the maximal temperature of 55°C ensured adequate coloration ($0.77 < \Delta E < 2.68$)

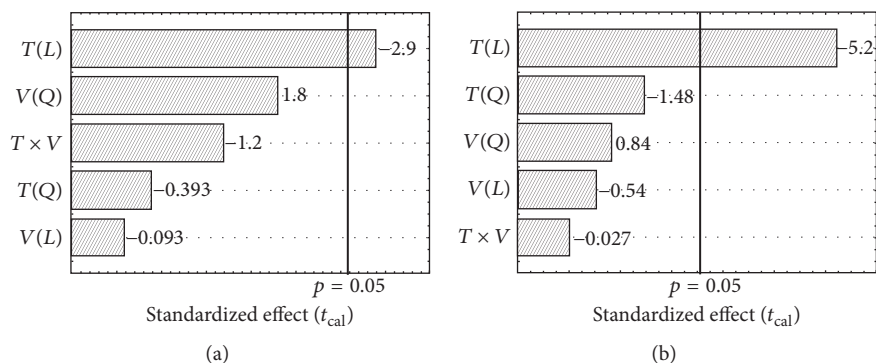


FIGURE 5: Pareto diagrams for process responses. (a) Extraction yield on dry basis; (b) monoterpene content of the essential oil from *S. terebinthifolius* fruits. $T(L)$: linear temperature effect; $T(Q)$: quadratic temperature effect; $V(L)$: linear velocity effect; $V(Q)$: quadratic velocity effect.

and suitable moisture content ($M^* < 12\%$). This drying temperature was close to the temperature of 60°C mentioned by De Carvalho et al. [31] for drying of *S. terebinthifolius* fruits for the production of pink peppercorns. Nonetheless, if the objective is extraction of volatile compounds, it is necessary to determine whether these drying conditions are sufficient to avoid losses of these compounds during drying.

3.4. Effects on Volatile Compounds. GC-MS analysis (Table 4) enabled detection of 25 compounds in the essential oil from *S. terebinthifolius* fruits. As shown in Table 4, 16 compounds were identified, representing, on average, approximately 98% of the chemical composition of the essential oil. The main compounds were α -phellandrene (14.26–20.46%), δ -3-carene (13.67–19.14%), limonene (15.19–18.28%), β -myrcene (8.12–12.75%), and germacrene D (6.25–10.90%). These results are similar, with slight variations, to those obtained by Barbosa et al. [32] and Cole et al. [12]. This variation observed in the same species collected at different locations (ecotypes) can often be explained by the high complexity of the chemical composition of essential oils, which depends on several factors, such as the location of harvest, climatic conditions, genetic variability (chemotype), pretreatment of the raw material (e.g., grinding, drying), and extraction process [3, 19, 33].

Figure 5 depicts Pareto diagrams showing the standardized effects of drying air temperature and velocity on the yield and monoterpene content of essential oil from *S. terebinthifolius* fruits, showing that air temperature had statistically significant negative effects on these parameters. These data indicate that an increase in air temperature manifests itself in a decrease in the extraction yield and monoterpene content, with a confidence level of 95%. Consequently, these results indicate a relationship between the volatilization of some volatile compounds from *S. terebinthifolius* fruits and the parameters of the drying process. It is worth mentioning that, in the literature, there are no studies that have addressed the influence of drying on essential oils from *S. terebinthifolius* fruits.

According to Figiel et al. [2], there are several different factors that can affect the loss of volatile compounds during

the drying process, for instance, the temperature reached, interactions between the volatiles and water vapor, or the hydrophobicity of the volatiles. Pinheiro [34] reported that the loss of volatile compounds during drying is based on the steam distillation operation (when a mixture of two immiscible liquids is distilled). Accordingly, during drying, some volatile components may have been carried away with water vapor within the fruit to the dryer outlet. In fact, Figiel et al. [2] found that, during vegetable drying, decreases in volatile concentration were strongly associated with the amount of water being evaporated. According to those authors, water acts as a solvent enabling diffusion of volatile compounds within the dried material, and water vapor acts as a carrier that enhances transfer of volatiles from the material being dried to the surroundings. In addition, during convective drying, volatile compounds are substantially exposed to oxidation processes due to the large volume of air flowing through the drying bed.

The dimensionless extraction yield and monoterpene content (relative to fresh fruits) for each assay (Table 1) are shown in Figure 6. The relative value was calculated in accordance with (5), with a relative value closer to 1.0 being more ideal in terms of drying performance. Figure 6 shows that the extraction yield and monoterpene content are close to 1.0 when drying is performed at mild temperatures (assays 1, 3, and 5). In contrast, drying at higher temperatures (assays 2, 4, and 6) decreases the extraction yield and monoterpene content, as compared with values obtained from fresh fruits. Therefore, drying at higher temperatures may reduce the extraction yield and monoterpene content of the essential oil from *S. terebinthifolius* fruits.

3.5. Optimal Drying Conditions. The coded models and the results of analysis of variance (ANOVA) are shown in Table 5. In this work, coded statistical models (quadratic polynomial equation) were used to predict responses in moisture content, total color difference, and monoterpene content as a function of drying air temperature. The adjustment quality of these models was evaluated by ANOVA; the experimental data were analyzed in STATISTICA software at a fixed significance level of 0.05. The data presented in Table 5 show that $F_{cal,1}$

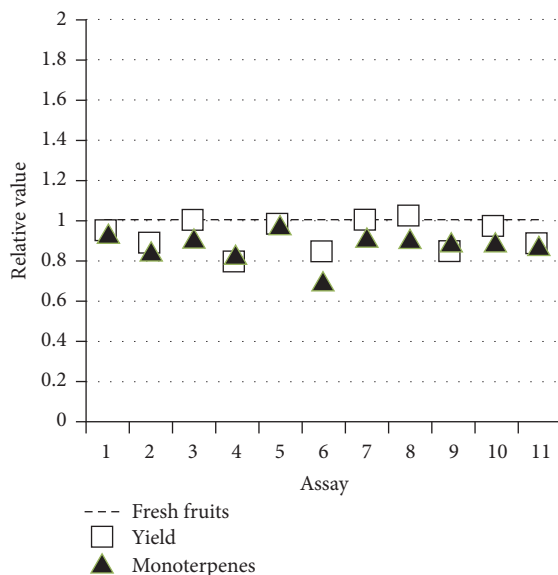


FIGURE 6: Dimensionless parameters (relative to fresh fruits) for each assay (from 1 to 11, under operating conditions of Table 1).

TABLE 5: Coded models and results of analysis of variance (ANOVA).

Model	Regression/residual		Lack of fit/pure error		R
	$F_{cal,1}$	$F_{tab,1}$	$F_{cal,2}$	$F_{tab,2}$	
$M^* = 6.48 - 3.43T + 0.675T^2$	41.78	4.459	0.7797	19.33	0.9553
$\Delta E = 1.80 + 2.24T + 1.65T^2$	22.40	4.459	1.072	19.33	0.9211
$MC = 73.3 - 5.67T - 2.23T^2$	20.27	4.459	12.64	19.33	0.9139

M^* : moisture content on wet basis; T : coded temperature according to Table 1; ΔE : total color difference; MC: monoterpene content; F_{cal} : F distribution (calculated); F_{tab} : F distribution (tabulated); R : correlation coefficient.

was greater than $F_{tab,1}$, $F_{cal,2}$ was less than $F_{tab,2}$, and the correlation coefficient (R) was greater than 0.9. Therefore, the results revealed that the models successfully described the effects of air drying temperature on the quality attributes of *S. terebinthifolius* fruits, under the operating conditions being studied (Table 1).

The response surfaces and contour plots are shown in Figures 7–9. These figures indicate that the quality attributes of *S. terebinthifolius* fruits depend on the operating conditions used for drying, especially the air temperature. The definition of the optimal operating conditions for the drying process depends on the target application of the product. Thus, if the objective is to obtain an essential oil with high monoterpene content (>70%), optimal conditions for drying are achieved at low air temperatures. As shown in Figure 9, for this purpose, temperatures in the range 35 to 55°C are recommended for drying. By contrast, if the objective is production of pink peppercorns, temperatures between 40 and 55°C are recommended for an optimal tradeoff between moisture content (Figure 7) and color (Figure 8).

Such conditions provide adequate moisture content ($M^* < 12\%$) without dramatic degradation of color for *S. terebinthifolius* fruits.

As mentioned above, the maximal temperature of 55°C was close to the temperature of 60°C mentioned by De Carvalho et al. [31] for the drying of *S. terebinthifolius* fruits for production of pink peppercorns. Karam et al. [3] reported that low drying temperatures generally have a positive influence on the quality of biological materials but require long processing times, which in turn have detrimental effects on product quality and increase costs. The authors emphasized that drying at temperatures of 55°C or 60°C can be considered an optimal compromise for the quality of dehydrated fruits/vegetables; under these temperature conditions, acceptable color and nutrient retention, relatively short drying time, and limited structural damage are achieved.

4. Conclusion

In the production of pink peppercorns, drying is undoubtedly the most important step. If drying is not performed properly, the quality of pink peppercorns decreases, affecting marketability. Appearance and flavor are among the most important quality criteria for pink peppercorns. In this work, a methodology for the assessment of pink peppercorn quality under different drying conditions was defined. The results

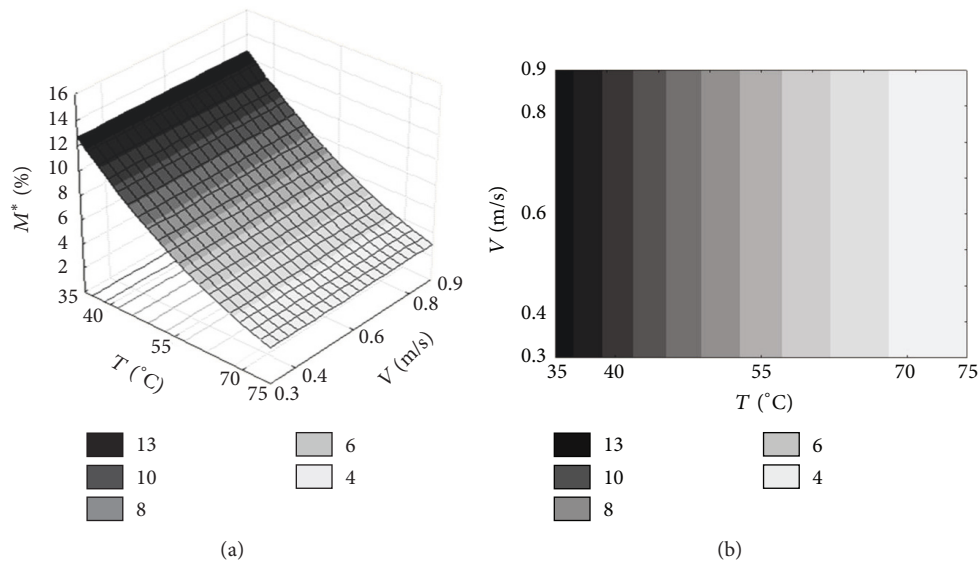


FIGURE 7: (a) The response surface and (b) a contour plot for moisture content on wet basis (M^*) as a function of air temperature (T) and air velocity (V).

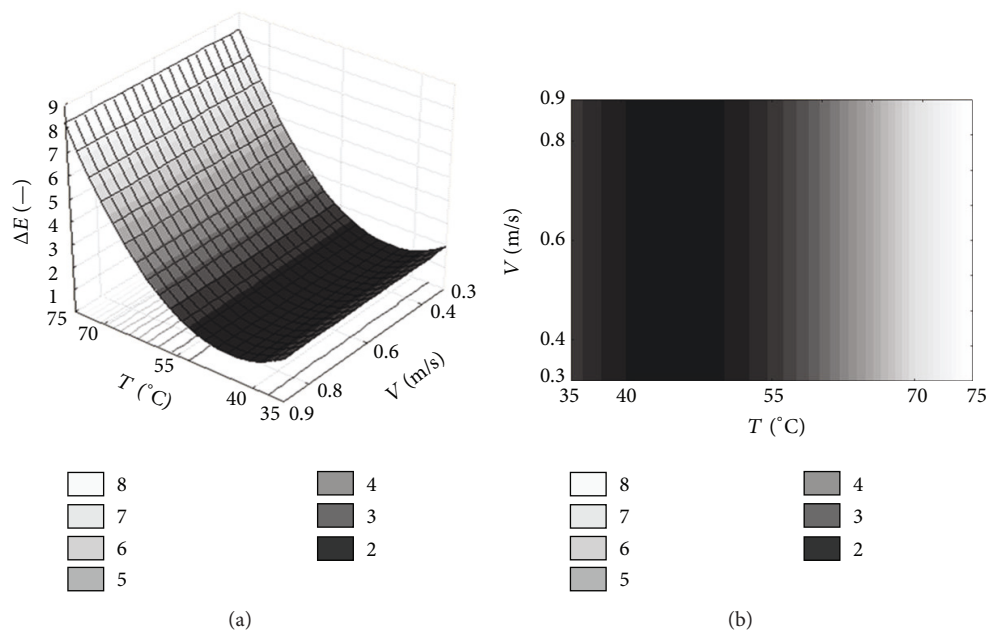


FIGURE 8: (a) The response surface and (b) a contour plot for the total color difference (ΔE) as a function of air temperature (T) and air velocity (V).

showed that drying in a tray dryer ensures integrity of the product. Air temperature had the greatest influence on the chemical and physical parameters that were investigated in this study, while air velocity had no statistically significant effects. Considering all the quality criteria, drying temperatures between 40 and 55 $^{\circ}\text{C}$ ensured the best compromise, in terms of adequate reduction of moisture without dramatic

degradation of color and essential oil. Further work is needed to devise a sensory analysis of pink peppercorns. Therefore, these results show the importance of monitoring drying conditions during the production of pink peppercorns. This approach enabled a better understanding of the phenomena involved and evaluation of the behavior of this material during the drying operation. In addition, this study can serve

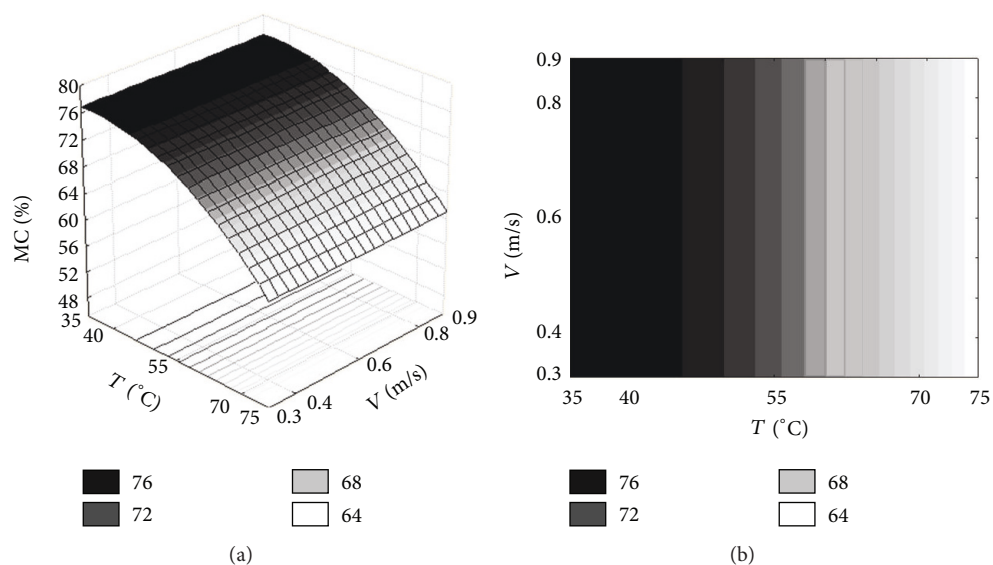


FIGURE 9: (a) The response surface and (b) a contour plot for monoterpene content (MC) as a function of air temperature (T) and air velocity (V).

as a basis for improving the quality of pink peppercorn product and thus meeting the quality requirements imposed by consumers.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

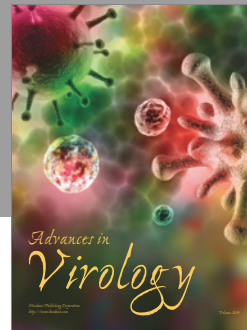
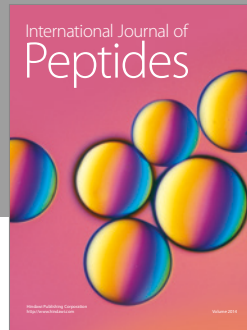
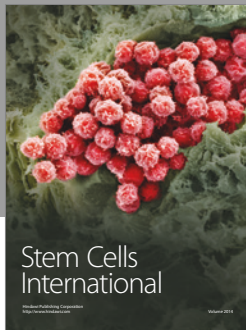
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