Article

Drying of *Phylla nodiflora* Leaves: Antioxidant Activity, Volatile and Phytosterol Content, Energy Consumption, and Quality Studies

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Abstract: Drying is an important process in the preservation of antioxidants in medicinal plants. In this study, leaves of *Phylla nodiflora*, or commonly known as frog fruit, were dried using convective drying (CD) at 40, 50, and 60 °C; vacuum-microwave drying (VMD) at 6, 9, and 12 W/g; and convective pre-drying followed by vacuum-microwave finish drying (CPD–VMFD) at 50 °C and 9 W/g. Drying kinetics of *P. nodiflora* leaves was modelled, and the influences of drying methods on the antioxidant activity, total phenolic content, volatile and phytosterol contents, energy consumption, water activity, and color properties were determined. Results showed that drying kinetics was best described by modified Page model. VMD achieved highest drying rate, whereas VMFD considerably reduced the drying time of CD from 240 min to 105 min. CPD–VMFD was the best option to dry *P. nodiflora* in terms of retaining volatiles and phytosterols, with lower energy consumption than CD. Meanwhile, VMD at 6 W/g produced samples with the highest antioxidant activity with 2,2'-Azinobis (3-ethylbenzthiazoline-6-sulfonic acid) (ABTS) and ferric reducing antioxidant power (FRAP) value of 11.00 and 15.99 µM Trolox/100 g dw, respectively.

Keywords: *Phylla nodiflora*; vacuum-microwaves; antioxidant activity; essential oil volatile composition; phytosterol; drying technology

1. Introduction

Antioxidants play important roles in nutrition by increasing the shelf life and preserving the nutritional aspect of lipid-containing food products. Antioxidants also prevent the oxidation of biological molecules in the body by chelating redox metals or quenching free radicals [1,2]. Plants contain numerous natural antioxidants and can be a source of new antioxidant compounds given the various chemical structures and mechanisms of action of these natural antioxidants [3]. *Phylla nodiflora* (L.) Greene, also known as *Lippia nodiflora* and widely known as frog fruit, belongs to family Verbenaceae and is distributed in subtropical and tropical regions [4,5]. Previous studies have confirmed the presence of antioxidants in *P. nodiflora* extracts [6,7], namely, stigmasterols, nodiflorethin, nodiflorin,

lippiflorin, and batalilfolin [4]. Traditionally, the leaves are used to treat diarrhea, fever, cold, knee joints pain, and gonorrhea [5]. Fresh leaves are high in moisture and are perishable commodities [8]. The antioxidant compounds in the leaves may undergo reduction during the period between harvesting and processing. Thus, fresh medicinal plants must be effectively preserved by removing moisture to inactivate enzymatic, chemical and metabolic reactions, which may otherwise cause the loss of important antioxidants [9].

Moisture removal from food is an integral operation in food processing. Convective drying (CD) has remained the most common drying method to reduce moisture from food and biomaterials. In CD, heat is transferred convectively from the hot air or combustion gasses to the cooler moist boundaries of the drying material. Thereafter, heat diffuses into the drying material primarily through conduction. Evaporation of moisture at the surface of the material is efficient; however, the removal of moisture from the center regions to the surface is slow because it is driven by a moisture concentration gradient, rendering CD a low drying rate method [10]. In view of the limitations of CD, there is increasing interest in emerging drying technologies, such as microwave drying, which gives better drying performance. Microwave drying is gaining popularity because drying time can be reduced and a higher retention of active constituents can be achieved. Despite fast drying rates, the heterogenic structure of materials and the inhomogeneity of microwave field may result in hot spots within the material at the final stage of drying [11]. A solution is to use microwave drying in combination with vacuum drying. This combination has been proven effective in many previous studies on preserving active compounds in the drying of herbs and fruits [12,13]. Furthermore, a lower pressure of vacuum-microwave drying (VMD) induces quicker water evaporation from food material at a lower temperature [14], thereby reducing chemical alterations [15]. However, high water evaporation from plant material at the initial stage of VMD can exceed the vacuum pump's capacity, so reducing the material load or a larger vacuum pump installation is required [11,14]. Pre-drying through CD, hereafter denoted as CPD, reduces the load mass of raw material to be dried in VMD. This two-stage drying method of CPD followed by vacuum-microwave finish drying (VMFD), hereafter denoted as CPD-VMFD, reduces the total cost of dehydration and improves the quality of products [16].

Although the chemical composition of several herb species dried using vacuum-microwaves has already been well recognized [13,17,18], to date no studies have investigated the influences of VMD and CPD-VMFD on the antioxidant activity of *P. nodiflora* and similar species. Therefore, it is of interest to evaluate the effectiveness of new vacuum-microwave drying methods compared to conventional CD in retaining bioactive constituents. In the present work, the drying kinetics and energy consumption of CD, VMD, and CPD-VMFD were determined, and the influence of all investigated drying methods was evaluated in terms of antioxidant activity, total phenolic content, concentration of volatiles and phytosterols, color properties, and water activity. Fatty acids were also profiled to identify active constituents in the plant extracts.

2. Results and Discussion

2.1. Drying Kinetics

Figure 1a shows the drying kinetics of *P. nodiflora* leaves dried with CD and CPD-VMFD, while Figure 1b shows the drying kinetics of *P. nodiflora* dried using VMD. Drying kinetics of all drying methods were best described by an exponential function, namely, the modified Page model (Equation (4)). Table 1 shows the values of both statistical parameters and mathematical constants of the Lewis, modified Page and Henderson and Pabis model. Selection of the best model was based on the highest Coefficient of determination (R^2) and the lowest root-mean-square error (RMSE), thereby suggesting goodness of fit. The values, R^2 and RMSE, obtained suggest a good agreement between the experimental values obtained and the modified Page model.

(a)

(b)

Figure 1. (a) Drying kinetics of *Phylla nodiflora* leaves dried using convective drying (CD) method at 40, 50, and 60 °C and vacuum microwave finish drying (VMFD) at 9 W/g preceded by convective predrying (CPD) at 50 °C. (b) Drying kinetics of *P. nodiflora* leaves dried using vacuum microwave drying (VMD) at 6, 9, and 12 W/g.

Table 1. Statistical parameters, total drying duration, and mathematical model constants describing the drying kinetics of *P. nodiflora* leaves.

Drying Condition	Model	<i>a</i>	<i>k</i>	<i>n</i>	<i>R</i> ²	RMSE	Drying Time (min)
CD 40 °C	Lewis	–	0.0138	–	0.9989	0.0113	360
	Page (modified)	0.9937	0.0126	1.0209	0.9989	0.0117	
	Henderson–Pabis	1.0000	0.0138	–	0.9989	0.0117	
CD 50 °C	Lewis	–	0.0246	–	0.9962	0.0216	240
	Page (modified)	0.9943	0.0151	1.1293	0.9989	0.0124	
	Henderson–Pabis	1.0284	0.0254	–	0.9972	0.0193	
CD 60 °C	Lewis	–	0.0287	–	0.9778	0.0493	180
	Page (modified)	1.0324	0.0371	0.9376	0.9788	0.0521	
	Henderson–Pabis	1.0168	0.0293	–	0.9782	0.0507	
VMD 6 W/g	Lewis	–	0.1860	–	0.9794	0.0534	21
	Page (modified)	0.9983	0.0766	1.4702	0.9996	0.0086	
	Henderson–Pabis	1.0402	0.1919	–	0.9813	0.0549	
VMD 9 W/g	Lewis	–	0.1860	–	0.9794	0.0534	21
	Page (modified)	0.9983	0.0766	1.4702	0.9996	0.0086	
	Henderson–Pabis	1.0402	0.1919	–	0.9813	0.0549	
VMD 12 W/g	Lewis	–	0.2769	–	0.9801	0.0523	14
	Page (modified)	0.9986	0.1403	1.4582	0.9996	0.0091	
	Henderson–Pabis	0.9986	0.1403	–	0.9996	0.0091	
CPD–VMFD 50 °C, 9 W/g	Lewis	–	0.0754	–	0.9811	0.0521	105
	Page (modified)	0.0733	0.7681	0.5011	0.9997	0.0007	
	Henderson–Pabis	0.0725	0.3734	–	0.9782	0.0049	

a—model constant; *k*—drying constant; *n*—dimensionless empirical constant; CD—convective drying; VMD—vacuum microwave drying; CPD—convective pre-drying; VMFD—vacuum microwave finish drying; *R*²—coefficient of determination; RMSE—root mean square error.

Drying duration of CD was the longest compared with VMD and CPD–VMFD. At the initial stage of CD, moisture loss was fast; however, successive moisture loss reduced with time. This phenomenon occurs because CD uses surface heating; the evaporation of moisture from the surface of plant material was efficient but moisture diffusion from internal region to the surface is slow and rate-limiting [19]. However, when VMFD at 9 W/g was applied to convective pre-dried leaves, drying time was 105 min, as opposed to 240 min of CD, thereby achieving 2.3 times shorter drying duration.

The relationship between drying rate and moisture ratio is shown in Figure 2. The drying rate of VMD was the highest, which was attributed to the volumetric heating of microwave drying. Microwaves penetrate inner regions of leaf tissue, thereby producing heat by the absorption of microwave energy by water molecules. Increased positive steam pressure in leaf structure pushes vapor to the surface to be removed [20]. CD recorded the lowest drying rates, but these values increased with increased temperatures. An increase from 40 to 50 °C showed an average increase of 0.003 min^{−1}, whereas in the case of VMD, an increase from 6 to 9 W/g showed a higher average drying rate of 0.012 min^{−1}. The low drying rate of CD at 50 °C can be compensated for by the introduction of VMFD, which showed a considerable improvement in drying rate at low moisture ratios.

Figure 2. Drying rate for *P. nodiflora* leaves dehydrated using CD method at 40, 50, and 60 °C, VMD at 6, 9, and 12 W/g, and VMFD at 9 W/g preceded by CPD at 50 °C.

The drying rate also decreased with decreased moisture ratio. This falling rate period is characterized by a continuous decrease in drying rate [17]. With VMD, a higher drying rate is typically observed at the initial stage as moisture content was initially higher in leaves, and this finding corresponded to a higher dielectric constant. Larger microwave radiation was absorbed by the sample, generating more heat, thereby increasing the drying rate. As drying progressed, the reduced amount of moisture reduced the microwave absorption, resulting in decreased drying rates.

2.2. Specific Energy Consumption

Profiles of specific energy consumptions are shown in Figures 3 and 4. Curve shapes revealed that as moisture content decreases, specific energy consumption increased, with a drastic increment at the final stage of drying, as characterized by the critical moisture content, K . The K values of all drying methods (below 0.1847 kg water kg⁻¹ dw) are indicated in Figures 3 and 4. The shape of characteristic curves resulted from the course of the drying kinetics shown in Figure 1a,b. The shape of the drying curves in Figure 1a,b shows that moisture removal was time consuming when moisture content was low. The prolonged time period to remove moisture at the final stages of drying corresponded to the high energy requirement for the removal of the same amount of moisture at the final stage of drying compared to initial drying stages. This behavior is usually seen in plant materials with osmotic cellular structure to limit the escape of moisture from plant tissues [21].

Figure 3. Profile of specific energy consumption for drying a gram of *P. nodiflora* leaves, dried using CD, VMD, and CPD-VMFD methods.

Figure 4. Profile of specific energy consumption related to the gram of water evaporated from sample of *P. nodiflora* leaves, dried using CD, VMD, and CPD-VMFD methods.

VMD required less energy to remove moisture compared with CD. However, energy consumption of CD can be minimized with VMFD, reducing final specific energy consumptions of CD by 80.2% and 51.1% (kJ/g water). CPD reduced water load by 92.4%, from an initial moisture content of 7.032 to 0.537 kg water kg⁻¹ dw. Drying intensity was also an influencing factor on the respective specific energy consumptions. Higher drying intensities in terms of increased hot-air temperature and microwave wattage reduced final specific energy consumptions (Figure 5), which is consistent with observations of past reports [21,22].

Figure 5. Final specific energy consumptions of CD, VMD, and CPD–VMFD of *P. nodiflora* leaves.

2.3. Antioxidant Activity and Total Phenolic Content Analysis

Analysis of antioxidant activity showed that the highest antioxidant activity was observed for VMD 6 W/g samples 2,2'-Azinobis (3-ethylbenzthiazoline-6-sulfonic acid (ABTS), 11.00 μ M Trolox/100 g dw; ferric reducing antioxidant power (FRAP), 15.99 μ M Trolox/100 g dw). The lowest antioxidant activity was obtained with VMD 12 W/g with an ABTS value of 6.62 μ M Trolox/100 g dw, whereas the lowest FRAP value of 8.26 μ M Trolox/100 g dw was shown by CD 40 °C samples (Table 2).

Table 2. Effects of drying methods on the antioxidant activity and total phenolic content of *P. nodiflora* leaves.

Drying Method	Antioxidant Activity (μ M Trolox/100 g dw)		Total Phenolic Content (mg/100 g dw)
	ABTS	FRAP	
Fresh	14.34 $\pm$ 0.93 ^b	18.25 $\pm$ 0.54 ^b	1484.46 $\pm$ 30.68 ^a
CD 40 °C	7.60 $\pm$ 0.46 ^{a,d}	8.26 $\pm$ 0.54 ^{a,c}	1609.73 $\pm$ 69.24 ^{a,c}
CD 50 °C	10.25 $\pm$ 0.77 ^{c,d}	14.18 $\pm$ 1.69 ^{e,f}	1762.20 $\pm$ 31.38 ^{a,c}
CD 60 °C	10.54 $\pm$ 1.22 ^c	14.17 $\pm$ 0.91 ^{e,f}	1828.92 $\pm$ 110.18 ^c
VMD 6 W/g	11.00 $\pm$ 0.19 ^c	15.99 $\pm$ 1.53 ^{b,f}	1661.54 $\pm$ 123.30 ^{a,c}
VMD 9 W/g	9.28 $\pm$ 0.34 ^{a,c,d}	10.21 $\pm$ 0.16 ^{c,d}	1091.26 $\pm$ 136.04 ^b
VMD 12 W/g	6.62 $\pm$ 0.63 ^a	9.25 $\pm$ 1.31 ^{c,d}	1473.23 $\pm$ 80.60 ^a
CPD–VMFD	7.11 $\pm$ 2.15 ^a	12.11 $\pm$ 0.50 ^{d,e}	2352.93 $\pm$ 101.09 ^d

ABTS—2,2'-Azinobis (3-ethylbenzthiazoline-6-sulfonic acid); FRAP—ferric reducing antioxidant capacity; VMD—vacuum microwave drying; CPD—convective pre-drying; VMFD—vacuum microwave finish drying; mean values with different alphabets (a, b, c, d) within each column is significantly different ($p < 0.05$), according to Tukey's test.

Active compounds of thermally dried herbs were assumed to have undergone thermal degradation and oxidative degradation in the case of CD and CPD–VMFD, thereby leading to the lower antioxidant activities observed. The results clearly indicate that a suitable application of hot-air temperature and microwave power, during CD and VMD, have the potential in yielding high antioxidant activities. A low microwave power of 6 W/g (VMD) and a higher temperature of 60 °C in CD were beneficial in yielding high antioxidant activities.

The intensity of drying treatment also affected antioxidant activity. The increase in temperature of CD, from 40 to 60 °C, shortened the drying length within 360 to 180 min. At 50 and 60 °C, the degradation of heat-sensitive compounds was considerably lower compared to CD at 40 °C, which was confirmed by relatively high antioxidant activity. These results indicate that the increase in temperature during CD is effective in accelerating the drying process, thereby shortening sample's exposure to elevated drying temperature in a highly oxygenated condition. With increased microwave power of VMD, from 6 to 12 W/g, antioxidant activity decreased. The increase in microwave power from 6 to 12 W/g decreased the drying time from 28 to 21 min, which; however, did not contribute to the preservation of antioxidant activity in the same way as observed in the case of CD. Evidently, the reduction in drying

time did not compensate for the degradation of the antioxidant compounds resulting from the greater amount of heat generated inside the material by increased microwave radiation.

The lengthy CD process can be shortened with microwave heating, as seen in CPD–VMFD. Results of FRAP assay showed that CPD–VMFD produced samples of higher or not significantly different antioxidant activity from samples dried exclusively with CD or VMD. The higher antioxidant activity of CPD–VMFD samples compared to CD at 40 °C (for FRAP assay) can be explained by the shortened drying time of the former. The presence of high moisture, oxygen, and heat during the lengthy drying process of CD 40 °C may have facilitated the degradation of antioxidant compounds by enzymes [23]. As for the higher antioxidant activity of CPD–VMFD samples compared with VMD at 9 and 12 W/g, the oxygen exposure during CPD–VMFD did not cause a large reduction in antioxidant activity. Several previous studies have indicated that phenolic content and antioxidant activity increase after heat treatment of peppermint leaves, *Artemisia annua* leaves [24], and culinary herbs, namely basil, rosemary, oregano, sage, thyme, and marjoram [25]. Wojdylo et al. suggested that polyphenols found in strawberry fruits that were exposed to oxygenated condition may have high antioxidant activity, although the increase is temporary [26].

Unexpectedly, the total phenolic content (TPC) values of fresh samples were lower than those of dried samples. The thermal treatment may have promoted an easier release of cell constituents from plant cells [27], as heat exerts modifications to plant tissue microstructure, thereby reducing cell integrity [28] and allowing for the easy exit of phenolic compounds from plant cells. Dried leaves have the tendency to exhibit increased bioactivity, as reported in past studies [29,30]. Moisture loss in leaves during drying, an abiotic stress, may have triggered the production and accumulation of phenolic compounds, thereby increasing the overall antioxidant activity [31]. Furthermore, the increase in antioxidant activity may have resulted from the Maillard reaction and the formation of Maillard reaction products (MRPs) with antioxidant power [26].

Although the TPC of samples dried with CPD–VMFD was high, antioxidant activity was relatively low. This finding implies that TPC of *P. nodiflora* did not correlate with the antioxidant activity. There are three major classes of chemical compounds in plants: phenolic metabolites, alkaloids, and terpenoids [32]. Phenolic compounds are the most researched and regarded as potent antioxidants; thus, the correlation between TPC and antioxidant activity has often been made. Terpenoids have also been established to exhibit strong antioxidant properties [33]. Therefore, in the case of *P. nodiflora*, it was assumed that terpenes and terpenoids present, which accounted for 36.5% of total volatiles, may have contributed to the overall antioxidant activity.

The overall antioxidant activity of *P. nodiflora* may be contributed by the major constituents in the extract and essential oil. For instance, 1-octen-3-ol, the major volatile compound (Table 3), β -sitosterol (Table 4) and α -linolenic acid, accounting for 48.3% of total fatty acids (Table 5), are among the antioxidants considered potent [34]. Terpenoids are known to exert antioxidant effect because of their ability to interact with free radicals. The presence of various terpenoids in the volatile fraction of *P. nodiflora* could also contribute to the antioxidant effect. With the presence of a wide variety of constituents in the extract of *P. nodiflora*, it can be assumed that the synergistic interactions between these molecules have effectively led to the observed antioxidant activities.

2.4. Analysis of Volatile Contents

Eighty-three compounds were identified in *P. nodiflora* leaves, amounting to 176.81 mg 100 g^{−1} db of volatile compounds in fresh leaves (Table 3). Figure 6 shows a typical chromatogram of the volatile compounds identified. The major volatiles present in fresh *P. nodiflora* were 1-octen-3-ol (60.34 mg 100 g^{−1} db), 2-hexenal (20.42 mg 100 g^{−1} db), and α -amorphene (10.55 mg 100 g^{−1} db). The volatiles present in *P. nodiflora* can be categorized under the following groups; alcohols (42.5%), sesquiterpenes (21.8%), aldehydes (16.2%), sesquiterpenoids (8.4%), monoterpenoids (3.2%), esters (3.0%), monoterpenes (2.5%) and diterpenoids (0.6%), thereby representing 98.2% of total volatiles. Other volatile compounds, phenylpropenes and ketones, were present in small concentrations.

Table 3. Concentration of volatile compounds influenced by various drying methods and conditions.

Compound	RT	Retention Indexes		Fresh	CPD-VMFD	CD			VMD		
						40 °C	50 °C	60 °C	6 W/g	9 W/g	12 W/g
		Exp.	Lit.			Concentration (mg 100 g ⁻¹ db)					
2-Hexenal	4.756	843	851	20.42	6.49	1.41	3.36	0.88	0.05	0.42	0.09
(E)-2-Hexen-1-ol	5.034	858	862	0.54	0.45	0.08	0.41	0.09	0.00	0.00	0.00
1-Hexanol	5.077	861	868	0.57	3.09	0.77	0.97	0.36	0.02	0.04	0.04
2-Heptanol	5.818	899	901	0.00	0.98	0.00	0.24	0.10	0.05	0.25	0.09
(E,E)-2,4-Hexadienal	6.083	909	911	0.93	0.51	0.15	0.40	0.11	0.00	0.00	0.06
Benzaldehyde	7.497	959	962	1.38	2.24	0.56	0.83	0.70	0.20	0.87	0.56
1-Octen-3-ol	8.099	883	880	60.34	79.06	19.30	21.84	21.11	1.89	8.80	6.03
6-Methyl-5-hepten-2-one	8.265	986	986	1.15	2.35	0.83	0.79	0.40	0.07	0.58	0.30
3-Octanol	8.444	992	994	0.64	0.33	0.04	0.03	0.06	0.01	0.22	0.05
6-Methyl-5-hepten-2-ol	8.528	997	994	3.49	5.19	1.66	2.22	1.50	0.10	0.90	0.36
Octanal	8.761	1002	1003	0.23	0.23	0.03	0.05	0.02	0.11	0.35	0.05
(E,E)-2,4-Heptadienal	9.012	1011	1012	0.22	3.93	0.38	1.42	0.93	0.16	1.36	0.69
(E,E)-2,4-Heptadienol	9.223	1015	-	0.91	0.02	0.03	0.05	0.01	0.00	0.01	0.00
<i>p</i> -Cymene	9.474	1024	1025	0.36	0.27	0.42	0.12	0.01	0.04	0.15	0.11
Limonene	9.610	1029	1030	0.35	0.10	0.13	0.20	0.00	0.02	0.47	0.14
Eucalyptol	9.700	1031	1032	0.01	0.00	0.00	0.00	0.00	0.00	0.02	0.00
Benzyl alcohol	9.893	1035	1036	1.01	1.07	0.12	0.04	0.24	0.04	0.25	0.02
β -Ocimene	9.893	1038	1037	0.12	0.34	0.03	0.06	0.09	0.01	0.04	0.01
Benzeneacetaldehyde	10.076	1042	1045	2.96	1.70	0.51	0.55	0.63	0.37	1.39	0.72
γ -Terpinene	10.563	1059	1060	0.49	0.15	0.04	0.16	0.03	0.00	0.02	0.11
cis-Sabinene hydrate	10.884	1068	1070	0.04	0.03	0.00	0.03	0.00	0.00	0.00	0.03
cis-Linalool oxide	11.065	1072	1074	2.33	6.46	0.90	2.41	1.94	0.21	1.40	0.00
Fenchone	11.584	1088	1096	0.02	0.00	0.02	0.00	0.01	0.00	0.02	0.67
2-Nonanone	11.669	1091	1092	0.00	0.20	0.12	0.54	0.56	0.00	0.10	0.00
Linalool	11.961	1100	1099	0.23	0.06	0.12	0.13	0.10	0.02	0.08	0.01
Nonanal	12.086	1104	1104	0.86	0.40	0.04	0.33	0.23	0.08	0.37	0.05
β -Thujone	12.184	1107	1114	0.05	4.09	0.13	0.81	1.22	0.15	1.56	0.22
Phenylethyl Alcohol	12.422	1114	1116	2.89	4.05	0.39	0.82	1.07	0.18	0.44	0.74
3-Thujanone	12.546	1118	1119	2.86	2.75	0.86	0.14	0.37	0.08	0.86	0.23
Camphor	13.500	1145	1143	0.78	0.76	0.11	0.19	0.20	0.03	0.28	0.46
Hexyl isobutyrate	13.609	1148	1150	0.12	0.02	0.07	0.03	0.00	0.00	0.02	0.16
Menthone	13.820	1154	1154	0.46	0.09	0.00	0.36	0.32	0.04	0.02	0.00
trans-Pinocamphone	13.900	1156	1160	0.12	0.21	0.20	0.13	0.07	0.03	0.23	0.02
Isomenthone	14.054	1161	1164	0.00	0.02	0.18	0.01	0.00	0.00	0.00	0.03
<i>p</i> -Menthan-3-one	14.166	1165	1166	0.55	0.13	0.07	0.02	0.00	0.00	0.01	0.00
Borneol	14.251	1167	1167	0.33	0.03	0.01	0.02	0.00	0.00	0.04	0.06
1-Nonanol	14.391	1171	1173	1.02	1.01	0.07	0.28	0.23	0.03	0.39	0.04
Menthol	14.474	1173	1175	0.00	0.02	0.09	0.08	0.01	0.02	0.01	0.20

Table 3. Cont.

Compound	RT	Retention Indexes		Fresh	CPD–VMFD	CD			VMD		
						40 °C	50 °C	60 °C	6 W/g	9 W/g	12 W/g
		Exp.	Lit.			Concentration (mg 100 g ^{−1} db)					
3-Pinane, cis	14.517	1175	1173	0.00	0.00	0.01	0.02	0.01	0.00	0.00	0.01
Terpinen-4-ol	14.639	1178	1177	0.00	0.03	0.00	0.01	0.00	0.00	0.00	0.00
<i>p</i> -Cymen-8-ol	14.903	1186	1183	0.36	0.40	0.01	0.12	0.08	0.02	0.15	0.00
Hexanoic acid butyl ester	15.070	1191	1189	0.41	0.01	0.01	0.21	0.10	0.01	0.22	0.13
Methyl salicylate	15.211	1195	1192	2.29	1.15	0.50	0.49	1.14	0.05	0.04	0.00
Estragole	15.336	1199	1196	1.52	1.55	0.17	0.57	0.56	0.10	0.59	0.27
2-Octynoic acid-methyl ester	15.558	1210	1212	0.29	0.75	0.09	0.30	0.22	0.07	0.35	0.17
Acetic acid, octyl ester	15.766	1223	1210	0.16	1.15	0.10	0.32	0.29	0.04	0.41	0.22
Isogeraniol	16.559	1235	1240	0.00	0.01	0.00	0.00	0.01	0.00	0.00	0.01
Butanoic acid, 2-methyl-, hexyl ester	16.628	1238	1236	0.04	0.00	0.00	0.01	0.09	0.00	0.00	0.01
D-Carvone	16.893	1245	1246	0.53	0.31	0.14	0.28	0.16	0.06	0.37	0.04
Isopentyl hexanoate	17.158	1253	1252	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00
Linalyl acetate	17.271	1257	1257	0.59	0.01	0.00	0.33	0.15	0.01	0.00	0.15
<i>iso</i> -3-Thujanol acetate	17.577	1265	1267	0.48	0.61	0.02	0.20	0.16	0.01	0.24	0.14
<i>neo</i> -3-Thujanol acetate	17.742	1271	-	0.05	0.00	0.04	0.00	0.00	0.00	0.03	0.00
Benzeneacetaldehyde, α -ethylidene-	17.826	1273	1279	0.34	0.21	0.10	0.00	0.01	0.02	0.12	0.03
<i>neo-iso</i> -3-Thujanol acetate	18.288	1280	-	0.09	0.01	0.01	0.01	0.01	0.00	0.00	0.00
Anethole	18.300	1287	1286	0.22	0.00	0.13	0.06	0.03	0.01	0.10	0.04
2-Undecanone	18.524	1293	1294	0.22	0.40	0.23	0.44	0.32	0.11	0.27	0.10
α -Cubebene	20.258	1357	1351	2.20	2.02	0.32	0.39	0.56	0.13	0.80	0.38
Propanoic acid	21.010	1378	1380	0.29	0.82	0.07	0.17	0.06	0.21	0.64	0.14
Copaene	21.137	1383	1376	5.37	4.26	0.50	0.70	1.41	0.34	1.87	0.74
β -Bourbonene	21.318	1390	1384	1.87	7.82	0.63	0.48	0.22	0.52	1.35	0.59
β -Cubebene	21.444	1391	1389	5.15	4.11	0.37	0.87	1.04	0.03	1.34	0.96
<i>cis</i> - α -Bergamotene	21.845	1409	1415	0.48	0.18	0.02	0.09	0.04	0.11	0.22	0.09
Caryophyllene	21.970	1417	1419	2.64	1.45	0.25	0.26	0.47	0.03	0.58	0.30
β -Duprezianene	22.190	1427	1423	1.83	0.22	0.06	0.12	0.01	0.41	0.13	0.65
β -Gerjunene	22.399	1437	-	0.19	0.14	0.01	0.02	0.03	0.00	0.03	0.03
Isogermairene D	22.523	1444	1448	0.08	0.00	0.00	0.00	0.00	0.00	0.00	0.01
Neoclovene	22.649	1450	1454	0.17	0.00	0.00	0.12	0.03	0.00	0.03	0.01
Alloaromadendrene	22.830	1457	1461	2.59	0.93	0.09	0.14	0.07	0.13	0.60	0.12
Sesquibabinene	22.886	1462	1464	3.93	1.30	0.19	0.17	0.12	0.19	0.94	0.33
9-epi-Caryophyllene	23.054	1470	1467	3.03	2.36	0.36	0.40	0.62	0.15	0.94	0.32
γ -Muurolene	23.275	1480	1477	0.11	0.06	0.01	0.00	0.02	0.00	0.01	0.01
γ -Curcumene	23.330	1483	1480	0.35	0.21	0.04	0.03	0.05	0.01	0.08	0.04
α -Amorphene	23.443	1489	1482	10.55	0.97	0.52	0.74	0.90	0.48	2.00	1.39
γ -Amorphene	23.665	1498	1496	2.20	1.63	0.39	0.33	0.54	0.15	0.76	0.56

Table 3. Cont.

Compound	RT	Retention Indexes		Fresh	CPD–VMFD	CD			VMD		
						40 °C	50 °C	60 °C	6 W/g	9 W/g	12 W/g
		Exp.	Lit.			Concentration (mg 100 g ^{−1} db)					
α-Muurelene	23.762	1505	1499	0.09	0.01	0.03	0.00	0.04	0.00	0.02	0.01
β-Bisabolene	23.874	1513	1509	0.21	0.02	0.01	0.05	0.02	0.00	0.07	0.08
δ-Cadinene	24.011	1522	1524	0.20	0.02	0.01	0.01	0.01	0.00	0.01	0.02
cis-Calamenene	24.151	1530	1531	5.53	5.71	0.99	1.01	1.71	0.28	1.70	0.92
α-Cadinene	24.319	1540	1538	2.11	0.73	0.13	0.11	0.21	0.08	0.31	0.20
α-Himachalene	24.433	1550	1549	1.01	0.13	0.03	0.03	0.03	0.04	0.13	0.07
Widdrol	25.413	1618	1610	0.05	0.08	0.00	0.01	0.01	0.01	0.03	0.03
Isopropyl myristate	27.409	1825	1827	0.28	0.31	0.07	0.12	0.08	0.33	0.61	0.25
TOTAL				176.81	179.02	44.47	57.83	53.32	16.15	48.02	29.94

RT—retention time; Exp.—experimental; Lit.—retention indices according to NIST 14 database; CD—convective drying; VMD—vacuum microwave drying; CPD—convective pre-drying; VMFD—vacuum microwave finish drying.

Figure 6. A typical gas chromatography—mass spectrometry (GC-MS) chromatogram of fresh *P. nodiflora*.

Leaves dried using CPD–VMFD (179.02 mg 100 g^{−1} db) achieved higher retention of volatiles compared with fresh leaves (176.81 mg 100 g^{−1} db), but the differences in values were marginal (Table 3). Thermal treatment of leaves during CPD–VMFD may have inactivated polyphenol oxidase (PPO), thereby reducing enzymatic degradation of volatiles [27]. In addition, short thermal treatment, achieved by the introduction of VMFD assured that volatiles were not exposed to high temperature for an excessively long period.

Consistent with the literature, CD and VMD led to considerable losses of volatiles, evidenced by the drying of thyme [17], basil [13], and rosemary [35]. In this study, CD retained a higher amount of volatiles than did VMD. A similar result was reported in the drying of rosemary [35]. In another study, VMD was shown to retain higher total volatiles because of the fast drying process at a mild temperature [18]. However, in this study, average temperature throughout VMD, irrespective of microwave power, was 46 °C. Slightly higher temperatures of CD at 50 and 60 °C and CPD–VMFD at 50 °C were advantageous in forming a partially dried surface layer on leaves that function as a barrier, thereby limiting the diffusion of volatiles and minimizing their loss [36]. Furthermore, the formation of a crust layer is characteristic of products dried using CD. Parameters in CD and VMD also affect the total concentration of volatiles. A moderate temperature of 50 °C and microwave power of 9 W/g were preferable in yielding higher total volatiles. At these conditions, leaves were subjected to moderate temperature at a relatively short time. It was suggested that VMD led to higher amount of volatiles loss as microwave treatment causes a more porous structure in plant tissue because of the high internal vapor formed during the drying process. The higher porosity of tissue structure of vacuum microwave-dried leaves, compared to convective-dried leaves, could be a possible reason that a higher amount of volatiles could escape to the surrounding medium [37,38].

Analysis of volatile compounds of dried samples showed that CPD–VMFD and CD 50 °C yielded the highest volatile concentrations. Therefore, both methods were used to determine the influence on phytosterols concentration. Table 4 shows the identified phytosterols in *P. nodiflora* and their respective concentrations. The total concentrations of phytosterols in fresh leaves were 1237.39 mg 100 g^{−1} db. β -Sitosterol was the major phytosterol in fresh and dried samples, followed by stigmasterol and campesterol. CPD–VMFD was able to retain a higher total yield of phytosterols (711.07 mg 100 g^{−1} db) compared with CD 50 °C samples (668.66 mg 100 g^{−1} db). Due to the chemical structures of phytosterols, such as the unsaturation at the side chain or ring, these compounds may undergo oxidation when exposed to air and heat treatment at high temperatures. The oxidation process of phytosterols is reported to increased when phytosterols are subjected to heat and the stability of phytosterols is also influenced by the temperature and time of heat treatment [39]. In this study, microwave treatment during VMFD benefited the retainment of higher phytosterol content. Lower temperature and reduced oxygen condition during VMFD may have minimized the thermo-oxidation of phytosterols, thereby increasing the stability of phytosterols. This result is in agreement with the results obtained by Soupas et al. [40], Rudzińska, Przybylski, and Wąsowicz [41], which all reported that phytosterol concentration decreased with increased drying temperature and prolonged drying time.

Table 4. Phytosterols profile of *P. nodiflora* dried using different drying methods.

Compound	Retention Time (min)		Fresh	CD 50 °C	CPD-VMFD
	Exp.	Lit.	Concentration (mg 100 g ⁻¹ db)		
β-Sitosterol	28.99	28.98	529.67	268.88	280.99
Stigmasterol	28.06	28.15	323.47	173.34	191.72
Campesterol	27.58	27.58	205.81	100.24	99.65
Betulin	31.16	31.17	30.68	37.30	39.17
Desmosterol	26.64	26.63	76.89	35.59	37.94
Cycloartenol	30.00	30.05	28.15	24.15	22.43
Lanosterol	26.89	26.88	40.58	19.88	20.31
α-tocopherol	25.97	25.95	2.14	9.30	18.86
Total	-	-	1237.39	668.66	711.07

Exp.—experimental; Lit.—Retention time according to Adams [42]; CD—convective drying; CPD—convective pre-drying; VMFD—vacuum microwave finish drying.

2.5. Fatty Acid Analysis

Thermal drying has little effect on the concentration of fatty acids because it was reported that fatty acids remained stable even at a high temperature of 325 °C [43]. In another study, less than 1% of long-chain fatty acids were lost when subjected to thermal hydrolysis at 90 °C for 30 min [44]. Therefore, drying temperatures of CD, VMD, and CPD-VMFD, applied in this study, can be assumed to have no degradative effect on fatty acids. Profiles of the relative abundance of fatty acids identified in *P. nodiflora* leaves are presented in Table 5. The major fatty acid present was α-linolenic acid, followed by palmitic acid, with boiling points of 231 and 351.5 °C, respectively [45,46]. Therefore, this further proves that the temperatures applied in this study could not have affected the total concentrations of α-linolenic acid and palmitic acid. α-linolenic acid is a plant-derived omega-3 fatty acid and is an antioxidant, widely researched for its role in mediating cardiovascular disease [47].

Table 5. Profile of fatty acid methyl esters of *P. nodiflora*.

Compound	Total Area (%)
α-linolenic acid	48.30
Palmitic acid	21.99
Linolelaidic acid	8.80
Oleic acid	7.98
Stearic acid	5.60
11-hexadecenoic acid, methyl ester, (11Z)-	1.53
Myristic acid	1.41
Behenic acid	0.71
Erucic acid	0.69
Arachidic acid	0.68
Palmitoleic acid	0.64
Elaidic acid	0.43
Heptadecanoic acid	0.26
cis-10-heptadecenoic acid	0.25
Lauric acid	0.22
Pentadecanoic acid	0.22
Capric acid	0.12
Linolelaidic acid	0.09
Tridecanoic acid	0.08

2.6. Color Analysis

Color values of fresh and dried *P. nodiflora* leaves are shown in Table 6. Parameters investigated include lightness (L^*), 0 (black) to 100 (white); greenness or redness (a^*), −60 (green) to 60 (red);

and blueness or yellowness (b^*), -60 (blue) to 60 (yellow). Color changes in herbs subjected to drying are usually due to the loss of green pigments and carotenoids or enzymatic browning [48]. However, the shaping of the color profile of the dried product depends not only on the time and drying conditions but also on the amount of residual water, which can be estimated by the water activity values.

Table 6. Color parameters of *P. nodiflora* leaves measured after applications of different drying methods.

Drying Conditions	Color Parameters		
	L^*	a^*	b^*
Fresh	46.90 ± 0.09^c	-6.33 ± 0.09^a	18.52 ± 0.12^a
CD 40 °C	$45.23 \pm 0.24^{a,b,c}$	-6.35 ± 0.07^a	15.92 ± 0.09^b
CD 50 °C	46.56 ± 0.13^c	$-5.5 \pm 0.04^{b,c}$	16.05 ± 0.13^b
CD 60 °C	$46.05 \pm 0.08^{b,c}$	-5.21 ± 0.03^c	15.86 ± 0.06^b
VMD 6 W/g	$45.62 \pm 0.16^{a,b,c}$	$-5.96 \pm 0.19^{a,b}$	17.17 ± 0.30^d
VMD 9 W/g	$44.54 \pm 0.04^{a,b}$	$-5.5 \pm 0.04^{b,c}$	16.14 ± 0.05^b
VMD 12 W/g	43.94 ± 0.22^a	-5.86 ± 0.05^b	$16.31 \pm 0.06^{b,c}$
CPD-VMFD	46.49 ± 0.16^c	-7.02 ± 0.02^d	$16.77 \pm 0.15^{c,d}$

CD—convective drying; VMD—vacuum microwave drying; CPD—convective pre-drying; VMFD—vacuum microwave finish drying; mean values with different alphabets (a, b, c, d) within each column is significantly different ($p < 0.05$), according to Tukey's test.

Fresh leaves recorded the highest L^* value, whereas VMD at 12 W/g resulted in the darkest colored leaves with lowest L^* value, but these values were not significantly different from those of VMD 6 and 9 W/g and CD 40 °C. These conditions promote PPO activity, which results in browning characteristics of dried products [49,50]. An increase in microwave power, from 6 to 12 W/g, associated with increased maximal temperature of dried material from 52 to 57 °C, decreased the L^* value from 45.62 to 43.94. At higher microwave powers, increased amounts of absorbed energy used to vaporize water were observed, which may have been sufficiently large to increase the sample temperatures further during the falling rate period, thereby producing darker leaves [38]. This finding was evidenced in a previous study, in which burnt spots were observed on mint leaves after VMD [38].

The loss in green color was assumed to be related to the loss of chlorophyll [48], as indicated by an increase in a^* values (Table 6) which is thought to be associated with decreased a_w (Table 7). This relationship was confirmed for CD and VMD samples because the decrease in a_w from 0.2252 to 0.1081 and from 0.2780 to 0.2353 resulted in increased a^* values from -6.35 to -5.21 and from -5.96 to -5.86 , respectively. The lowest values of a^* were -6.35 and -7.02 , which were found for CD 40 °C and CPD-VMFD samples, respectively. In the case of CD 40 °C, the processing temperature was the lowest, whereas combined drying CPD-VMFD underwent at a relatively short time and moderate conditions in terms of temperature and microwave power, thereby leading to lower extents of chlorophyll degradation.

Table 7. Water activity of different drying methods.

Drying Method	Water Activity, a_w
Fresh	0.9879 ± 0.006^a
CD 40 °C	$0.2252 \pm 0.003^{b,d}$
CD 50 °C	0.1485 ± 0.010^c
CD 60 °C	0.1081 ± 0.010^c
VMD 6 W/g	0.2780 ± 0.001^d
VMD 9 W/g	$0.1743 \pm 0.044^{b,c}$
VMD 12 W/g	$0.2353 \pm 0.008^{b,d}$
CPD-VMFD	0.1339 ± 0.020^c

CD—convective drying; VMD—vacuum microwave drying; CPD—convective pre-drying; VMFD—vacuum microwave finish drying; mean values with different alphabets (a, b, c, d) within each column is significantly different ($p < 0.05$), according to Tukey's test.

Carotenoids, which tend to have yellowish color, were far better preserved in VMD 6 W/g, as indicated by a higher b^* value. High vacuum conditions and lower temperatures of VMD compared with CD methods guaranteed improved preservation of carotenoids. In the presence of oxygen, the susceptibility of carotenoids to oxidation is high [51]; therefore, we can assume that higher oxidation occurred during CD. However, shortening drying time of CD by applying vacuum microwaves increased the value of b^* coordinate, thereby indicating the effectiveness of CPD-VMFD in preserving natural colorants.

2.7. Water Activity Analysis

Water activity (a_w) measures the extent of water removal and the amount of residual moisture of dried products [10]. To eliminate the possibility of microbial growth and unfavorable enzymatic activities, dried products should have low a_w , ranging from 0.60 to 0.80. Spoilage bacteria are inhibited below a_w of 0.91 and most types of mold are inhibited at a_w of 0.80 [51]. However, for dried leaves or tea products, the suggested a_w should be below 0.267 [52]. The lowest a_w recorded was for convective-dried (CD) samples at 60 °C (Table 7). Leaves dried at CD 50 and 60 °C and CPD-VMFD have significantly lower a_w , because drying temperatures coupled with extended drying durations of CD ensured minimal residual moisture in leaves. Increasing the hot-air temperature during CD corresponded to lower a_w , because the temperature difference between the leaf material and surrounding heated air increased, which resulted in greater heat and moisture transfer. Thus, higher evaporation of water occurs, leaving material with a lower amount of moisture. The low a_w values obtained for all drying methods suggest that samples were microbiologically stable, and that a limited amount of residual water was present for undesirable chemical reactions to occur.

3. Materials and Methods

3.1. Chemicals and Reagents

2,2'-Azinobis (3-ethylbenzthiazoline-6-sulfonic acid (ABTS)), potassium persulfate, 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox), ferric reducing/antioxidant power (FRAP) reagent, acetate buffer, acetic acid, 2,4,6-tripyridyl-1,3,5-triazine (TPTZ), Folin-Ciocalteu reagent, sodium carbonate, gallic acid, *p*-methoxyphenol, chloroform, potassium hydroxide, hexane, sodium chloride, methanol, hydrochloric acid, boron trifluoride-methanol solution (BF_3/MeOH), magnesium sulfate, pyridine, and *N,O*-Bis(trimethylsilyl)trifluoroacetamide (BSTFA)) were purchased from Sigma-Aldrich (Steinheim, Germany).

3.2. Plant Material Preparation

Fresh leaves of *P. nodiflora* were purchased from TKC Nursery (Negeri Sembilan) and identified at the Forest Research Institute Malaysia with voucher number (049/17). Leaves were cleaned and vacuum-oven dried at 60 °C for 24 h to obtain the initial moisture content as determined by Equation (1).

$$\text{Initial moisture content} = \frac{\text{Initial weight} - \text{dry matter weight}}{\text{dry matter weight}} \quad (1)$$

3.3. Drying Methods

Drying was conducted in the Institute of Agriculture Engineering (Wroclaw, Poland). Leaves were dried in 40 g portions using three methods, namely CD, VMD, and CPD-VMFD. Drying was ended when the mass loss of consecutive samples was 0.05 g or less. For VMD and CPD-VMFD, the sample temperature was measured with an infrared thermometer (Flir i50, Portland, OR, USA) after removing from the dryer, thereby providing an indication of the maximal temperature.

3.3.1. Convective Drying (CD)

Leaves were dried at 40, 50, and 60 °C in a convective hot-air oven designed and constructed in the Institute of Agriculture Engineering (Wroclaw, Poland). Leaves were spread on a wire mesh tray and placed on top of drying chambers with an air velocity of 1.8 ms⁻¹.

3.3.2. Vacuum-Microwave Drying (VMD)

Leaves were dried in a vacuum-microwave dryer (Plazmatronika, Wroclaw, Poland) at microwave powers of 6, 9, and 12 W/g. Leaves were placed in an organic glass container, connected to a vacuum system that was rotated at a speed of 6 rpm. The pressure in the vacuum drum was varied from 4 to 6 kPa. An electric fan at the bottom of the dryer provided an air stream velocity and temperature of 1 ms⁻¹ and 22 °C, respectively, to prevent local overheating of leaves.

3.3.3. Two-Stage Drying—Convective Pre-Drying Followed by Vacuum-Microwave Finish Drying (CPD-VMFD)

CPD of leaves was conducted with the same procedures as CD at 50 °C for 90 min to achieve a moisture content of 0.537 kg water kg⁻¹ dw. Pre-dried leaves were finish dried in a vacuum-microwave dryer (Plazmatronika, Wroclaw, Poland) at 9 W/g, with similar operating conditions to those for VMD. Intermediate hot-air temperature at 50 °C and microwave wattage at 9 W/g were used in CPD-VMFD, as both parameter levels ensured the highest concentration of volatile compounds in the dried product (Table 3).

3.4. Modelling of Drying Kinetics

Drying kinetics were determined based on the mass losses of leaves throughout the drying process. Drying curves were plotted as functions of moisture ratio against time. Moisture ratios *MR* were determined from Equation (2):

$$MR = \frac{M(t) - M_e}{M_0 - M_e} \quad (2)$$

where *M(t)* indicates moisture content after drying time *t*, *M*₀ denotes initial moisture content and *M*_{*e*} denotes equilibrium moisture content, determined at the final stage of the drying process as an asymptotic value of the function fitted to the experimental points using Table Curve 2D Windows v2.03 (Jandel Scientific Software, San Rafael, CA, USA). To select the most suitable model, three commonly used thin layer models, namely Lewis (Equation (3)), modified Page (Equation (4)), and Henderson and Pabis (Equation (5)) were examined.

$$MR = \exp(-k \cdot t) \quad (3)$$

$$MR = a \cdot \exp(-k \cdot t^n) \quad (4)$$

$$MR = a \cdot \exp(-k \cdot t) \quad (5)$$

where *MR* is moisture ratio, *a* is model constant, *k* denotes drying constant, and *n* represents the dimensionless empirical constant.

3.5. Determination of Energy Consumption

3.5.1. Energy Consumption of CD

Energy consumed during CD, *E*_C (kilojoules) was determined as follows [53]:

$$E_C = \left(\frac{N_f}{6} + N_h \right) \times t \quad (6)$$

where N_f (kilowatts) is the power consumption of the fan supplying air to the six pipes, fixed with an electric heater of power consumption N_h (kilowatts), whereas t is the time of drying (seconds).

3.5.2. Energy Consumption of VMD

Energy consumed during VMD, E_{VM} (kilojoules) was determined as follows [53]:

$$E_{VM} = \left(\frac{N_M}{\eta_M} + N_V + N_e \right) \times t \quad (7)$$

where N_M is the output power of magnetrons (kilowatts), η_M is the efficiency of magnetrons, N_V is the power consumption of vacuum pump (kilowatts), N_e is the power consumption of electric engine rotating the container (kilowatts), and t is drying time (seconds).

3.5.3. Specific Energy Consumption of CD, VMD, and CPD-VMFD

Specific energy consumption is calculated to determine the energy consumption per unit mass of fresh weight and energy consumption per unit mass of water. The specific energy consumption expressed in kJ/g fresh weight of sample can be useful in prediction of energy expenditure for drying of specified plant material. On the other hand, specific energy consumption expressed in kJ/g water contained in sample enables comparison of energy consumption for different plant materials with different moisture content.

Specific energy consumptions of CD, E_{Cm} ($\text{kJ g}^{-1} fw$), and E_{CW} ($\text{kJ g}^{-1} \text{water}$) were calculated based on Equations (8) and (9) [53], and the specific energy consumptions of VMD, E_{VMm} ($\text{kJ g}^{-1} fw$) and E_{VMW} ($\text{kJ g}^{-1} \text{water}$), Equations (10) and (11) [53], were calculated as (1) ratio of energy consumption to the initial sample mass, m (grams); and (2) ratio of energy consumption to the mass of water W (grams) removed from the sample.

$$E_{Cm} = \frac{E_C}{m} \quad (8)$$

$$E_{CW} = \frac{E_C}{W} \quad (9)$$

$$E_{VMm} = \frac{E_{VM}}{m} \quad (10)$$

$$E_{VMW} = \frac{E_{VM}}{W} \quad (11)$$

Specific energy consumptions for CPD-VMFD, E_{C-VMm} ($\text{kJ g}^{-1} fw$) and E_{C-VMW} ($\text{kJ g}^{-1} \text{water}$), are as shown in Equations (12) and (13), respectively [53]. Both equations are the ratios of the sum of energy E_C and E_{VM} to the mass of initial sample, m (grams) and total mass of water (grams) from CPD, W_C and VMFD, W_{VM} removed from the sample, respectively.

$$E_{C-VMm} = \frac{E_C + E_{VM}}{m} \quad (12)$$

$$E_{C-VMW} = \frac{E_C + E_{VM}}{W_C + W_{VM}} \quad (13)$$

3.6. Extraction of Polyphenol Compounds

Ground samples (0.3 g) were measured into tubes. Then, 80% aqueous methanol (0.7 mL) was added with 1% HCl and stirred gently. Tubes with suspension were sonicated twice for 15 min and left at 4 °C for 24 h. The extract was centrifuged for 10 min at 15,000 rpm (MPW-360R, Warsaw, Poland) and supernatants were collected.

3.7. Antioxidant Activity Analysis

3.7.1. ABTS^{•+} Radical-Scavenging Assay

ABTS assay was conducted as previously described by Re et al. [54]. ABTS was dissolved in water to form a concentration of 7 mM. ABTS stock solution was mixed with 2.45 mM potassium persulfate to obtain ABTS radical cation (ABTS^{•+}). This mixture was left to stand for 12–16 h in the dark at room temperature. The solution was diluted with distilled water to obtain an absorbance of 0.700 ± 0.02 at 734 nm. Diluted radical solution (3 mL) was mixed with 20 μ L of the extracted supernatant. Absorbance was determined using a UV-vis spectrophotometer (Shimadzu, UV-2401 PC, Kyoto, Japan) at 734 nm after 6 min. Results were expressed as micromolar Trolox per 100 grams of dry weight (dw).

3.7.2. FRAP Assay

FRAP assay was performed as described previously by Benzie and Strain [55]. FRAP reagent was prepared by mixing acetate buffer (300 μ M, pH 3.6), solution of 10 μ M TPTZ in 40 μ M HCl and 20 μ M of FeCl₃ (Sigma-Aldrich, Steinheim, Germany) at a ratio of 10:1:1 (v/v/v). The FRAP reagent (300 μ L) and sample solution (10 μ L) were mixed thoroughly. Absorbance was read at 593 nm using a UV-vis spectrophotometer (Shimadzu, UV-2401 PC, Kyoto, Japan) after 10 min. A standard curve was constructed using different concentrations of Trolox.

3.8. Analysis of Total Phenolic Content

Total phenolic content was determined using the Folin–Ciocalteu method, previously described by Gao, Ohlander, Jeppsson, Björk, and Trajkovski [56]. Leaf extract (0.1 mL) was mixed with 2 mL of water and 0.2 mL of Folin–Ciocalteu reagent. The mixture was incubated at room temperature for 3 min and added with 1 mL of 20% sodium carbonate. Absorbance was measured using a UV-vis spectrophotometer (Shimadzu, UV-2401 PC, Kyoto, Japan) at 765 nm after 1 h of incubation at room temperature. Quantification was determined based on a gallic acid standard curve. Results were expressed as gallic acid equivalence (in milligrams per 100 grams of dw).

3.9. Analysis of Volatile Compounds

Analysis of volatile compounds was conducted using headspace solid phase microextraction (HS-SPME). Approximately 0.25 g of crushed dried and fresh leaf samples were placed in vials with the internal standard; *p*-methoxyphenol (2 μ g). Vials were covered with PTFE/silicon septum and placed in a water bath at 60 °C for equilibration (10 min). SPME fiber (Supelco, Bellefonte, PA, USA) with a divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS, 50/30 μ m, coating 2 cm) (Supelco) was used. Prior to extraction, SPME fiber was conditioned by inserting into a GC injection port for 1 h at 220 °C. For each extraction, SPME fiber was inserted into a vial that contained a sample kept in a water bath at 60 °C for 30 min. Next, the fiber was drawn into a needle and injected into the injection port of a GC-MS at an injection temperature of 220 °C for 3 min. The GC-MS analysis was performed on a Varian CP-3800/Saturn 2000 (Varian, Walnut Creek, CA, USA) equipped with a capillary column, ZB-5MS (30 m $\times$ 0.25 mm i.d $\times$ 0.25 μ m film thickness) (Phenomenex, Shim-Pol, Poland).

Our gradient temperature program was as follows. The initial oven temperature was 50 °C, which was increased to 130 °C at a rate of 4 °C min^{−1}. Then, the temperature was raised to 180 °C at 10 °C min^{−1} and finally raised up to 280 °C at a rate of 2 °C min^{−1}. Samples were injected in split mode (1:10), and helium gas was used as the carrier gas at a flow rate of 1.0 mL min^{−1}. Mass spectra were obtained in EI ionisation mode at 70 eV within a scan range of 35–550 m/z. After each extraction, SPME fiber was prepared for the subsequent extractions by placing in the injection port of GC-MS for 15 min at 220 °C, to ensure complete elution of volatiles. Identification of all volatile constituents was based on a comparison of experimentally obtained compound mass spectra with mass spectra

available in NIST14 database. Furthermore, the experimentally obtained retention index (RI) by Kovats was compared with RI available in the NIST WebBook and literature data [42].

3.10. Extraction of Lipids

Lipids were extracted using a previous method by Folch, Lees, and Stanley [57]. Lipids from ground leaves were extracted using 2:1 chloroform–methanol (*v/v*) for 24 h and filtered thereafter. The resultant filtrate was subjected to vacuum rotary evaporator at 65 °C to remove extraction solvent. Crude lipids obtained were weighed and later saponified (10 min at 80 °C) with 10 mL of 0.5 M KOH/MeOH, boiled with reflux for 5–10 min and left to cool at 12 °C. The sample was placed in a separation funnel; hexane (10 mL) and water (10 mL) were added sequentially twice and mixed vigorously. To facilitate phase separation, 5 mL of saturated NaCl solution was added, and the mixture was mixed vigorously and left for phase separation. After separation, the bottom layer (fatty acids) was separated and removed from the top layer (sterols).

3.11. GC-MS Analysis of Phytosterols

Phytosterol portion was dried with MgSO_4 and evaporated using a vacuum rotary evaporator to remove the extraction solvent. The resultant residue was added with 0.2 mL of pyridine and 0.2 mL of BSTFA (silylation agent) in a capped tube. Cholesterol (1.0 mg, Sigma-Aldrich, Steinheim, Germany) was added as the internal standard. The reaction was heated at 60 °C for 45 min in an incubator orbital shaker at 110 rpm. Once derivatization was completed, the mixture was transferred to small capped tubes and subjected to GC-MS analysis. Scanning was performed at an EI of 70 eV, at a mass range of 50–400 m/z , and with a 5-scan s^{-1} mode. The carrier gas used was helium with a flow rate of 1.0 mL min^{-1} at a split ratio of 1:10. The temperature gradient programs used was as follow: (a) 45 °C for 3 min, (b) 45 °C to 220 °C at a rate of 5 °C min^{-1} , and (c) 220 °C to 250 °C at a rate of 10 °C min^{-1} and, lastly, maintained at 250 °C for 2 min. The injector was held at a temperature of 260 °C. Identification of plant sterols was based on the comparison of experimentally obtained mass spectra with ones available in literature and with relative retention times of standards. The concentrations of identified phytosterols were obtained by comparing the peak area of each compound to the peak area of the internal standard, cholesterol, with a concentration of 1 mg/mL.

3.12. GC-MS Analysis of Fatty Acids

The fatty acid portion was acidified with 1 M HCl and extracted with 10 mL of hexane. The organic portion was separated and subjected to a vacuum rotary evaporator to remove the extraction solvent. Next, the sample was subjected to methylation (10 min, 80 °C) by adding 4 mL of 14% BF_3/MeOH (*v/v*). Fatty acid methyl esters (FAMES) that were formed were extracted using 2.5 mL of hexane, dried with MgSO_4 , and filtered using a cotton plug and silicate power into a small container. The FAME profile was analyzed using GC-MS (GCMS-QP 2020, Shimadzu, Kyoto, Japan). Separation was conducted using the Zebron ZB-WAX capillary column (30 m $\times$ 0.25 mm i.d. $\times$ 0.25 μm film thickness) (Phenomenex, Torrance, CA, USA). GC-MS analysis was conducted as follows: Scanning was performed from 50–400 m/z in electronic impact (EI) at 70 eV, in the 5-scan s^{-1} mode. Helium was used as the carrier gas with a flow rate of 1.0 mL min^{-1} and a split ratio of 1:10. The temperature gradient program used was (a) 45 °C for 3 min; (b) 45–220 °C with a rate of 5 °C min^{-1} ; (c) 220–250 °C at a rate of 10 °C min^{-1} and kept at 250 °C for 2 min. An injector was held at 260 °C.

3.13. Color Analysis

The colors of fresh and dried powdered *P. nodiflora* leaves were determined with Minolta a Chroma Meter CR-200 (Minolta Co. Ltd., Osaka, Japan). Color data were expressed as CIE L^* , a^* , b^* coordinates. Color measurements were measured five times to obtain a mean value per sample.

3.14. Water Activity Analysis

The water activity of fresh and dried leaves was determined with a water-activity meter (Aqualab 4TE, Pullman, WA, USA). Fresh and ground leaves were placed and spread up to three-fourths of the sample cup which was inserted into the measuring chamber. Measurement of water activity was carried out at an average temperature of 24.9 ± 0.05 °C.

3.15. Statistical Analysis

Results are presented as mean $\pm$ standard deviation. One-way analysis of variance (ANOVA) was conducted using SPSS 20 (IBM, Tulsa, OK, USA). Significant differences between means were evaluated using Tukey's test at a significance level of $p \leq 0.05$. Mathematical modelling of the drying kinetics of CD, VMD, and CPD-VMFD methods was achieved with Table Curve 2D Windows v2.03 (Jandel Scientific Software, San Jose, CA, USA). R^2 RMSE were used to evaluate the goodness of fit of models (Equations (3)–(5)) to experimental data. All determinations were performed in triplicate.

4. Conclusions

The drying kinetics of *P. nodiflora* leaves were best described by the modified Page model. VMD achieved the shortest drying time, whereas CD was the most time-consuming method. However, the application of VMFD, in CPD-VMFD reduced drying time of CD considerably. The application of CPD-VMFD is also justified by the lower specific energy consumption compared with conventional CD. CPD-VMFD reduced the final specific energy consumptions by 80.2% and 51.1% (kJ/g water) compared with CD 50 °C. Moreover, CPD was effective in reducing water content by approximately 92.4%, from the initial moisture content of 7.032 to 0.537 kg water kg⁻¹ dw of samples. Based on the antioxidant-activity results, VMD at 6 W/g showed the highest antioxidant activity, consistent for both ABTS and FRAP analyses. β -Sitosterol was the major fatty acid that may have contributed to the antioxidant activity of *P. nodiflora*. Water-activity results showed that all methods produced samples that were microbiologically and shelf stable. Color analysis revealed that CPD-VMFD was superior in preserving the color of *P. nodiflora*. CPD-VMFD is a promising drying method based on the results of TPC, volatile, and phytosterol analysis.

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