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The Stability of Color and Antioxidant Compounds in Paprika (*Capsicum annuum* L.) Powder During the Drying and Storing Process

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Abstract The objectives of this study were to examine changes in the color and antioxidant compounds of paprika powder under various conditions, as well as to establish the suitable conditions for drying and storage. Paprika was dried using the following methods: freeze-drying, vacuum drying, far infrared-ray drying, and hot-air drying. Measurements of the moisture content, color pigments, and antioxidant compounds (total carotenoids, capsanthin, ascorbic acid, and total polyphenols) were completed during 120 days of storage at 4 and 30°C. We found that drying methods, storage temperatures, and packaging materials affected the American Spice Trade Association (ASTA) and Hunter color values, as well as the antioxidant content of paprika powder. There was a high correlation ($r=0.87$, $p<0.01$) between the ASTA color and the a*/b* value. The loss of red color was closely related to the reduction of moisture content ($r=0.81$, $p<0.01$) during storage. Drying paprika with a low temperature in the absence of air resulted in better retention of the carotenoids and ascorbic acid. Also, as the retention of the carotenoids and ascorbic acid increased, the stability of the red pigment increased. Freeze-drying was found to be the most suitable drying method for the stability of the antioxidant compounds and red pigment.

Keywords: paprika powder, antioxidant, drying method, color, stability, storage

Introduction

Recent epidemiological studies have associated the use of antioxidant vitamin supplements with substantial reductions in cancer and coronary heart disease risks (1-4). Fresh fruits and vegetables are the best sources of antioxidant vitamins (5), and the ripe fruit of red paprika is well known for being a good source of antioxidant compounds, as well as a natural food colorant due to its high carotenoid pigment content.

Paprika is commonly used as a food coloring product in its powdered and oleoresin forms. The carotenoid molecules in paprika are highly unsaturated, and their stability during drying and storage is very important for making a final food product attractive and acceptable in terms of its color and nutritive value (6, 7). The primary red carotenoid in paprika is capsanthin, which accounts for 30-60% of the total carotenoids (8). Capsanthin is regarded as a functional material because it has singlet oxygen-quenching abilities (9). Carotenoids are relatively stable when they are within intact plant tissues, but once processed, carotenoids are isolated and vulnerable to heat, light, and air (10).

Studies show that paprika tends to lose its red color when exposed to air. With this color loss there is a simultaneous decrease in antioxidant vitamins such as vitamin C and E, and the various carotenoids (11, 12). Also, additional studies indicate that temperature, environmental humidity, light, and packaging containers may be other factors for the degradation of color pigment (7, 13, 14). Malchev *et al.* (12) postulated that the stability of the major carotenoids in red pepper during storage is affected by drying conditions and the degradation rate. The high temperature and long drying period associated with conventional hot-air drying (HD) often cause heat damage to vegetables (15, 16). This is why vacuum drying (VD) at temperatures below 30°C is a preferred method for dehydrating heat-sensitive products (17). Furthermore, VD and freeze-drying (FD) methods are often applied to obtain high-value products since they reduce damage from heat and oxidation.

Several studies indicate that color degradation in paprika can be prevented by using natural antioxidants such as rosemary extract, tocopherols, and ascorbic acid (18, 19). However, there have been insufficient studies on the drying and storage conditions that would allow for maximal preservation of the antioxidant compounds and minimal degradation of the pigments.

Therefore, we designed this study to evaluate changes in the color and antioxidant compounds of paprika powder under various conditions, as well as to establish the most suitable conditions for drying and storage.

Materials and Methods

Samples and reagents Fresh paprika was obtained locally in Haman, Korea. All the reagents used were of HPLC grade. For other purposes, analytical grade reagents were filtered through a 0.45 μm membrane before use.

Preparation of samples and drying methods The pericarps of fresh paprika were taken and sliced to 3 mm. The initial moisture content of the fresh paprika was determined to be 92.7%; it was dried using the following various methods: a vacuum oven, a freeze drier (Bondiro; Ilshin Lab Co., Ltd., Gyeonggi, Korea), a far-infrared ray drying oven (SLD-1400S; CILIC, Gyeonggi, Korea), and a HD oven (FO-600M; Jeio Tech, Daejeon, Korea). The drying conditions of the vacuum oven and freeze drier were 40°C at 20 mmHg and -45°C at 50 mmHg,

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respectively. The far-infrared ray drying (FIRD) and HD were both carried out at air temperatures of 50 and 80°C and 1.2 m/sec air velocities. In a preliminary experiment, the drying was completed until final product moisture of approximately 14% was reached via regular interval checks with an infrared-ray moisture meter (F-1A; Kett, Fukuoka, Japan). The dried paprika was ground by a mill (SQ-107; Il-jin, Gyeonggi, Korea) in order to pass through an 0.85 mm sieve, and then it was packaged by nylon or a low density polyethylene film (LDPE). The thickness and oxygen transmission rate of the nylon film were 0.015 mm and 52.6 $mL/m^2 \cdot 24$ hr, respectively. Those of LDPE were 0.06-0.065 mm and 380-470 $mL/m^2 \cdot 24$ hr, respectively. All the samples were stored in an incubator at 4 and 30°C for 4 months. Measurements of the moisture content, color, and antioxidant compounds (total carotenoids, capsanthin, ascorbic acid, and total polyphenols) were completed at 0, 7, 30, 60, 90, and 120 days.

Moisture content and color determination The change in moisture content during storage was determined using a 105°C air oven and AOAC methods (20). The extractable color was measured by the American Spice Trade Method 20.1. (21), and the color intensity of the extract was measured with a UV-VIS spectrophotometer (Shimadzu, Kyoto, Japan) set at 460 nm. The following equation was then applied:

$$\text{ASTA (American Spice Trade Association) color value} = \frac{\text{Absorbance} \times 16.4}{\text{Sample (g)}}$$

The apparent color was measured by a colorimeter (CM-3500d; Minolta, Tokyo, Japan) and expressed as the L^*(lightness), a^*(redness), and b^*(yellowness) values of the Hunter color system.

Analysis of total carotenoids A method by Choi and Ha (22) was employed to determine the total carotenoid content. The pigments were extracted by blending the samples with acetone in a homogenizer. The residue was re-treated with acetone and the procedure was repeated until the filtrate was colorless. Na_2SO_4 was added, and the remnants from filtration and dehydration were treated with 60% KOH/methanol. This unsaponifiable fraction was defined as the total carotenoids. The total carotenoid (TC) content was calculated from the equation below.

$$\text{Carotenoid (mg\%)} = \frac{\text{Absorbance} \times \text{Volume} \times 1{,}000}{E_{\text{cm}}^{1\%}\ (2{,}400) \times \text{Sample (g)}}$$

Analysis of capsanthin The analysis of capsanthin was performed using a method previously described by Mingueg-Mosquera and Hornero-Mendez (23). A 1.5 g portion of paprika was extracted in a homogenizer (Ultra-turax; Ika Werke, Staufen, Germamy) with 50 mL of acetone until the extracts were colorless. All the extracts were pooled in a separating funnel and treated with 100 mL of ethyl ether. The ether phase containing the carotenoids was saponified with 100 mL of 20%(w/v) KOH/methanol. The saponified carotenoids were then extracted with ethyl ether. The ether layer was washed with distilled water, dried over anhydrous Na_2SO_4, and the solvent evaporated. The carotenoids were recovered in acetone and filtered through a 0.45 μm filter for analysis by HPLC (1100 series; Hewlett Packard, Palo Alto, CA, USA). The capsanthin was separated on a 150×4.6 μm (i.d.) column packed with a Pinnacle II C_{18} (Restek, PA, USA) 5 μm phase and eluted with 78:22 (v/v) acetone-water. The flow rate was 1 mL/min and detection was carried out at 450 nm.

Analysis of ascorbic acid The measurement of ascorbic acid (AA) was performed using a method adapted from Han *et al.* (24). Each sample (2 g) was homogenized with 5 mL of 5% metaphosphoric acid and filtered through a 0.45 μm filter. The AA was separated on a 150×4.6 mm (i.d.) column packed with a Pinnacle C_{18} 5 μm phase and eluted with 100% KH_2PO_4. The flow rate was maintained at 1 mL/min and detection was carried out at 254 nm.

Analysis of total polyphenols Here we followed a method that was previously presented by Benvenuti *et al.* (25). A 1 g amount of paprika powder was extracted in a homogenizer with 20 mL of methanol/HCl 2% (95:5 v/v) and centrifuged for 15 min at 10,000×g. The solution was diluted to volume with methanol/HCl 2%. Then 5 mL of Folin Ciocalteau's reagent and 10 mL of saturated sodium carbonate solution (75 g/L) were mixed with 1 mL of the sample extract. The solution was brought to 100 mL with water, and the absorbance was measured at 750 nm. The results were expressed as mg of garlic acid per g of fresh paprika.

Statistical analysis All the collected data were analyzed by analysis of variance (ANOVA) and the Duncan's multiple range test using the programmed SPSS (version 11; SPSS Inc., Chicago, IL, USA). The significance level was $p<0.05$ unless otherwise indicated. The Pearson's correlations between the color and antioxidant compounds and between the ASTA color and moisture content were also determined.

Results and Discussion

The effects of drying and storage conditions on moisture content The initial moisture content in red pepper is very important with regards to its pigment stability (26). Previous studies showed that a higher initial moisture level in red paprika powder resulted in less change in the color value and inhibited pigment destruction (27, 28). In this experiment, the average moisture content of the paprika powder was 14.4%. In all treatments the moisture content decreased with an increasing storage period (0-120 days) (Table 1). The moisture slightly decreased to a 11.68-10.23% range during the 120 day storage period at 4°C. However, it dramatically shifted to 9.82-4.25% during the same period at 30°C. Overall, drying and storing paprika at high temperatures seemed to bring significant moisture loss. Also, there was an antagonistic effect between storage temperature and moisture content. Finally, Paprika powder

Table 1. Moisture content in paprika powder packed with nylon film and LDPE film using different drying methods at 0 and 120 days storage (%)

Drying method[1)]	Drying time (hr)	0 day	120 day, Nylon		120 day, LDPE	
			4°C	30°C	4°C	30°C
FD	72	$14.59^{a2)}$	11.21^{c}	9.31^{b}	10.23^{c}	8.32^{c}
VD	10	14.51^{a}	11.07^{c}	9.16^{b}	11.01^{b}	8.74^{b}
FIRD 50	5.8	14.45^{a}	11.68^{b}	9.20^{b}	11.37^{a}	8.99^{a}
FIRD 80	5	13.64^{b}	11.62^{b}	5.75^{c}	10.52^{c}	4.25^{e}
HD 50	12	14.65^{a}	11.66^{a}	9.82^{a}	11.21^{ab}	8.32^{c}
HD 80	10	14.28^{a}	11.59^{a}	5.35^{d}	10.95^{b}	4.82^{d}

[1)] FD, Freeze drying; VD, vacuum drying; FIRD 50, far-infrared ray drying at 50°C; FIRD 80, far-infrared ray drying at 80°C; HD 50, hot-air drying at 50°C; HD 80, hot-air drying at 80°C.

[2)] Means with the same letter in a column of each sample are not significantly different over an increasing storage period (p<0.05).

packed with nylon film appeared to retain its moisture better than with LDPE.

Evaluation of color The values for the apparent and extractable colors of paprika powder are shown in Fig. 1 and 2, respectively. The apparent color was expressed as the L^*, a^*, and b^* values of the Hunter color system. The extractable color was measured by acetone extraction following the ASTA method.

For the apparent color, the L^*, a^*, and b^* values were significantly different among the various drying methods. The FD showed a higher L^* value than the other methods, but the a^* value of FD was similar to VD. A high drying temperature of 80°C decreased all the color values notably. Paprika dried with FIRD at 80°C and HD at 80°C turned out to be a darker, more brownish color than the samples dried at 50°C. When paprika powder was stored at 30°C it became darker. The b^* value showed a slight increase with 120 days of storage. Overall, the Hunter color values were affected more by the drying temperature than by the storage temperature or packaging materials. In the freeze-dried samples, storage at 4°C provided the least amount of pigment loss, which is in agreement with Cinar (7). The Δ E values were not significantly different among the samples (data not shown).

The ASTA color is generally used to determine paprika quality in manufacturing and trade. Depending on the drying methods and storage temperatures, the ASTA values here showed distinct losses of red color after 120 days of storage. At day 0 for storage, the highest ASTA value was with HD at 80°C, followed by VD > HD at 50°C > FIRD at 80°C > FIRD at 50°C > FD, indicating a different trend compared to the Hunter color values. Drying methods such as VD and HD that required relatively longer drying times (more than 10 hr) under increased heat produced higher ASTA values compared to the FIRD method, which had a shorter drying time at the same drying temperature. This suggested that more browning occurred during the longer drying process with the heating temperatures, thus affecting the ASTA color values. This is probably related to the Maillard reaction caused by the appreciable amounts of reducing sugars and amino acids in paprika (29). There was a large decrease in ASTA values for the paprika powder stored at 30°C. This phenomenon was even more obvious when the paprika powder was dried at 80°C. At a storage temperature of 4°C, the ASTA value of paprika packaged with nylon film was slightly higher than the value for paprika packaged with LDPE. This was opposite to the ASTA value that resulted at 30°C. The effects of the storage temperatures and packaging materials on paprika's color stability were more marked for the ASTA color values than the Hunter color values.

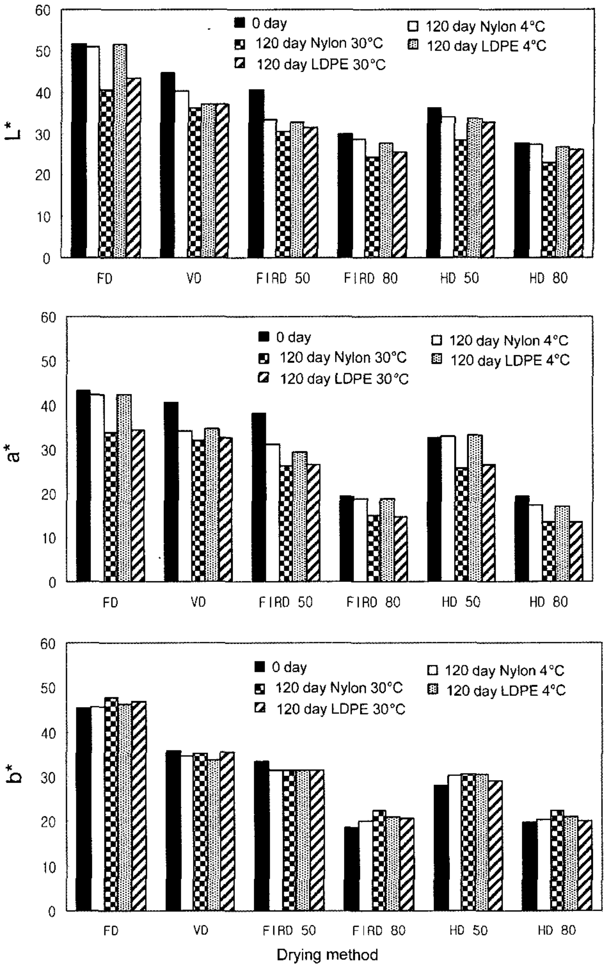


Fig. 1. Change in Hunter color values[1)] of paprika powder packaged with nylon and LDPE films using different drying methods at 0 and 120 days storage. FD, Freeze drying; VD, vacuum drying; FIRD 50, far-infrared ray drying at 50°C; FIRD 80, far-infrared ray drying at 80°C; HD 50, hot-air drying at 50°C; HD 80, hot-air drying at 80°C. [1)] L^*: Measures lightness and varies from 100 for perfect white to zero black; a^*: measures redness when plus and greenness when minus; b^*: measures yellowness when plus and blueness when minus. Values are means±SD, n=3.

The a^* value and ASTA value indicating red color appeared to be related to the moisture content. We found that the greatest amount of moisture was lost when paprika powder was dried by FIRD at 80°C or HD at 80°C, and then stored at 30°C. The color values of these samples decreased severely due to the destruction of red pigments such as carotenoids, which included capsanthin (6). The

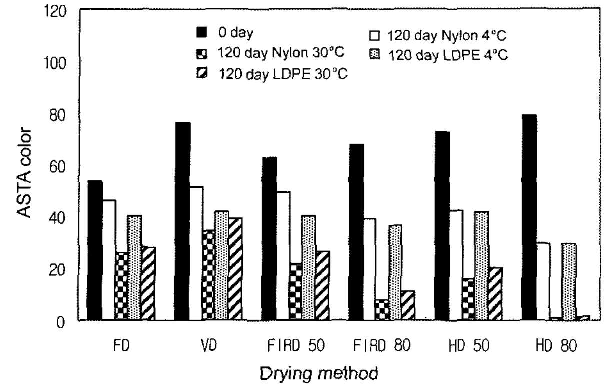


Fig. 2. Change in ASTA color of paprika powder packaged with nylon and LDPE films using different drying methods in 0 and 120 days. FD, Freeze drying; VD, vacuum drying; FIRD 50, far-infrared ray drying at 50°C; FIRD 80, far-infrared ray drying at 80°C; HD 50, hot-air drying at 50°C; HD 80, hot-air drying at 80°C. Values are means±SD, n=3.

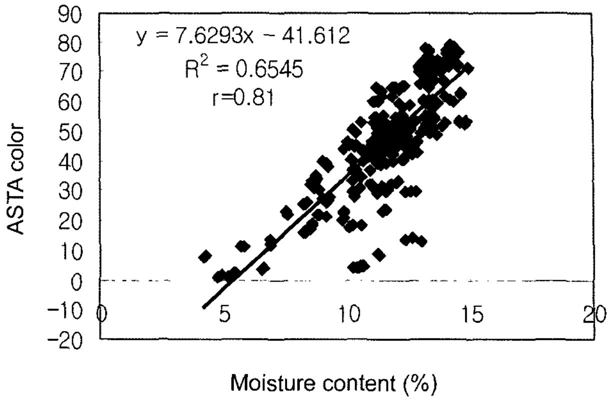


Fig. 3. Correlations between moisture content and ASTA color during storage.

correlation between the ASTA color and the moisture content was particularly high ($r=0.81$, $p<0.01$), as shown in Fig. 3. It is probable that a greater quantity of water in the paprika powder helped hinder oxygen access to the carotenoids (30). Therefore, when paprika powder is stored, the relative humidity or packaging materials must be considered along with the moisture content.

The ASTA color was highly correlated with a^*/b^* ($r=0.87$, $p<0.01$), but not with the a^* value or a^*/L^* (Table 2). Hong and Bae (31) stated that an a^* value measured with a colorimeter indicated redness; however, the a^*/b^* ratio represented the redness of red pepper very well, even though it generally indicates redness and yellowness.

Retention of antioxidant compounds The antioxidant content of the fresh paprika control, and the paprika powders dried with various drying methods are shown in Table 3. The fresh paprika contained the highest amount of antioxidant compounds such as TC, capsanthin, AA, and polyphenols. Each drying process destroyed the antioxidant compounds differently. The paprika dried by FIRD at 50°C and HD at 50°C, retained higher TC than when dried by FD, VD, and FIRD at 80°C and HD at 80°C. It seems that the retention of the TC in paprika powder was affected by factors other than drying temperature and air. Paprika dried by VD and FD retained high amounts of capsanthin with 8,961.43 and 7,380.74 μg/g, respectively.

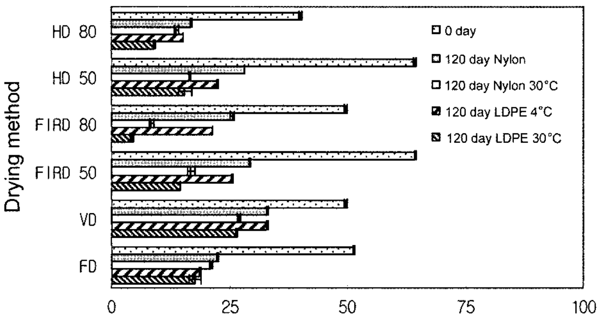
The amounts of capsanthin retained with FIRD at 50°C, HD at 50°C, FIRD at 80°C, and HD at 80°C were similar, ranging from 5,752.32 to 6,281.08 μg/g immediately after drying. A high amount (1,730.90 μg/g) of AA, which was 85.8% of the AA in fresh paprika, was retained in paprika powder samples dried by FD. The paprika dried by VD retained the second greatest amount of AA at 53.5% (1,080.06 μg/g). However, most of the ascorbic acid was destroyed at a high drying temperature (80°C) in both HD and FIRD. This was apparently due to AA oxidation in the presence of heated air. Each type of drying process destroyed more than 92% of the total polyphenols in fresh paprika. By nature paprika has extremely small quantities of total polyphenols (4.56 mg/g) compared to the total polyphenol content (65.8-106.2 mg/g) of green tea (32). The major phenolic losses that typically occur during processing are brought about by oxidative enzyme actions, such as those of polyphenoloxidases and peroxidases (33). Cutting the fresh paprika before drying may have largely reduced the amount of phenolics by enzymatic oxidation. Also, when grinding the dried paprika more destruction may have occurred.

As shown in Fig. 4A, the greater the drying and storage temperatures, the greater the loss of TC. The paprika samples stored at 4°C retained a higher amount of TC than those stored at 30°C, for all treatments. Carnevale *et al.* (34) stated that the loss of red color in paprika was caused by autoxidation of the carotenoids. According to Malchev *et al.* (12), the stability of the primary carotenoids during storage is dependent on the drying conditions and the

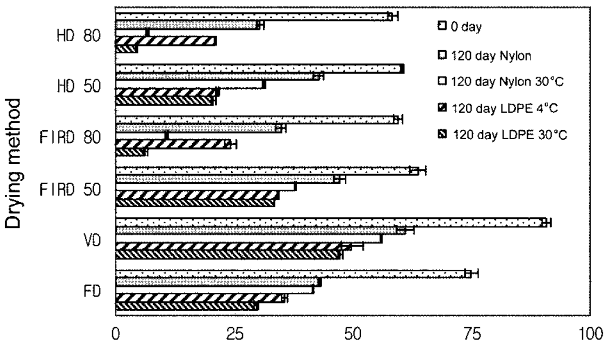
Table 2. Correlation coefficients between colors and antioxidant compounds of paprika powder

	Total carotenoid	Capsanthin	Ascorbic acid	a^*	a^*/b^*	a^*/L^*
Capsanthin	0.85$^{+1)}$					
Ascorbic acid	0.47	0.61				
a^*	0.49	0.66	0.73^{+}			
a^*/b^*	0.81^{+}	0.75^{+}	0.31	0.56		
a^*/L^*	0.42	0.53	0.33	0.81^{+}	0.66	
ASTA color	0.87^{+}	0.87^{+}	0.43	0.51	0.87^{+}	0.44

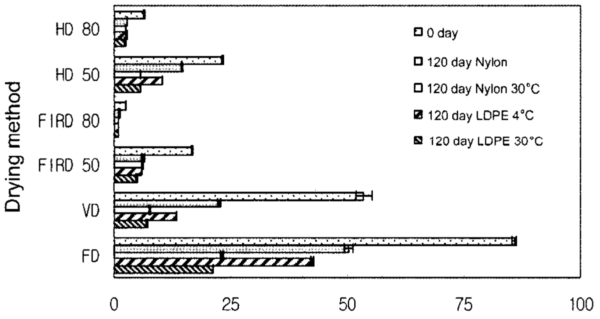
$^{1)+}$Significant at $p<0.01$.



(A) The retention of total carotenoid (%)



(B) The retention of capsanthin (%)



(C) The retention of ascorbic acid (%)

Fig. 4. The retentions of antioxidant compounds in paprika powder packaged with nylon and LDPE films using different drying methods at 0 and 120 days of storage. FD, Freeze drying; VD, vacuum drying; FIRD 50, far-infrared ray drying at 50°C; FIRD 80, far-infrared ray drying at 80°C; HD 50, hot-air drying at 50°C; HD 80, hot-air drying at 80°C.

% Retention

$$= \frac{\text{the amount of antioxidant compound in paprika powder}}{\text{the amount of antioxidant compound in fresh paprika}} \times 1$$

Values are means±SD, n=3.

degradation rate increases as the drying temperature increases, which partly agrees with our results.

Although capsanthin is a carotenoid, the effects of the various drying methods on its retention were not the same as those found for TC (Fig. 4B). When paprika samples that were dried by HD or FIRD at 80°C were stored at 30 °C, the retention of capsanthin was less than 4-6%, whereas the capsanthin in samples dried by both drying methods at 50°C, and then stored at 30°C, was retained to 20.7-37.8%. In a study by Kim *et al.* (35), the capsanthin contents of red pepper samples stored at 0 and 20°C were 88.6 and 60.9% of the initial contents, respectively, during 6 months of storage. At a high storage temperature of 30°C, the loss of AA was severely accelerated compared to the losses of TC and capsanthin (Fig. 4C). According to Aleman and Navarro (36), pigment degradation in paprika coincides with the destruction of AA and vitamin E, and continues with the degradation of the carotenoids due to their oxidation in air. Lavelli *et al.* (37) reported an 88% loss in AA when tomatoes were dried at 80°C for 7 hr to a 10% moisture content. The AA of paprika dried by FD was retained at 85.8%. However, after 120 days of storage only 50.4% was retained at 4°C and 23.1% at 30°C, when stored with the nylon film. This suggests that the drying temperature at which paprika powders are heated in the presence of air is the most important factor affecting AA retention. In addition, the temperature and amount of air transmitted through the packaging film are important concerns during storage.

Table 3. Comparison of antioxidant compounds in paprika powder using various drying methods

Drying method[1]	Total carotenoid (mg%)	Capsanthin (μg/g)	Ascorbic acid (μg/g)	Total polyphenol (mg/g)
FP	6409.52$^{a2)}$	9854.68^{a}	2016.68^{a}	4.56^{a}
FD	3294.86^{c}	7380.74^{c}	1730.90^{b}	0.34^{b}
VD	3186.29^{d}	8961.43^{b}	1080.06^{c}	0.23^{c}
FIRD 50	4129.14^{b}	6281.08^{d}	337.47^{d}	0.21^{c}
FIRD 80	3179.43^{d}	5854.03^{e}	49.33^{e}	0.20^{c}
HD 50	4123.05^{b}	5956.77^{e}	470.32^{d}	0.23^{c}
HD 80	2561.52^{e}	5752.32^{e}	129.11^{e}	0.19^{c}

[1] FD, Freeze drying; VD, vacuum drying; FIRD 50, far-infrared ray drying at 50°C; FIRD 80, far-infrared ray drying at 80°C; HD 50, hot-air drying at 50°C; HD 80, hot-air drying at 80°C; FP, fresh paprika.
[2] Means with the same letter in a column for each sample are not significantly different with an increasing storage period (p<0.05).

Most of the total polyphenol content in the paprika powder was destroyed by the drying process. Hence, it was difficult to find the effect of the storage conditions on polyphenol loss (data not shown).

We found that the red pigment and amount of antioxidant compounds were positively correlated (Table 2). A high retention of ascorbic acid coincided with high values of a*, giving high correlation coefficients (r=0.73, p <0.01), but ascorbic acid retention did not coincide with the ASTA values. There was also a high correlation coefficient (r=0.87, p<0.01) between the capsanthin, or TC, and the ASTA color.

A high retention of antioxidant compounds could be obtained by employing drying methods at low temperatures in the absence of air. According to Lin *et al.* (16) and Tijskens *et al.* (38), AA is relatively unstable to heat, oxygen, and light and the retention of this nutrient can be used as an indicator for the quality of dried vegetable slices. When AA is retained well, other nutrients are also likely to be preserved.

In conclusion, drying methods, storage temperatures, and packaging materials affected values of ASTA color and Hunter color, as well as the antioxidant content of paprika powder. During the storage period, the paprika packaged with nylon film and stored at 4°C had high color and antioxidant stability. Therefore, the use of a packaging container that prohibits the transmission of air or moisture is recommended. FD was the most suitable drying method for maintaining the quality of the paprika powder. However, the long drying time that it required remains a shortcoming. FIRD at 50°C was a better drying process than HD at 50°C, in terms of a shorter drying time and the antioxidant and red pigment stability. Therefore, when the antioxidant activity of paprika powder is applied in food products, the overall antioxidant activity should be examined as well as any interactions of the antioxidant compounds.

Acknowledgements

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