

# Effect of Water Activity and Temperature on the Color Change of Red Pepper (*Capsicum annuum* L.) Powder

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**Abstract** Isotherm characteristics of red pepper powder and the effect of temperature and water activity ( $A_w$ ) on its color change were investigated. Monolayer moisture contents of red pepper powder decreased from 0.1218 to 0.0912 g water/g solid with increasing temperature from 25 to 50°C. The color change of red pepper powder was greatly dependent on temperature and  $A_w$ . As temperature and  $A_w$  increased, red color of pepper powder increasingly faded out to become brown and tarnish black, which is mainly attributed to the degradation of carotenoid pigments and development of browning compounds. Color parameters such as Hunter- $L$ ,  $a$ ,  $b$  values and other color functions as well as browning index and ASTA color values represent color changes of red pepper powder as influenced by temperature and  $A_w$ .

**Keywords:** red pepper, color, water activity, temperature, hunter color function

## Introduction

Red pepper (*Capsicum annuum* L.) is extensively used through the world as a natural food colorant or a seasoning agent due to its attractive red color, unique sweet taste, and pungency. As the most important agricultural product in Korea with the market value of about one billion dollars a year, it is widely used as a main ingredient in traditional Korean foods such as *gochujang* and kimchi (1,2).

Color is the most important quality attribute of red pepper products that determines their overall quality and consequently their final market price (1,3,4). The characteristic color of red pepper is due to ketocarotenoid compounds such as capsanthin, capsorubin, and cryptocapsin (1,5). Capsanthin is the major red pigment, which represent as much as 50% of the total carotenoids, followed by capsorubin (6). Other pigments are xanthophylls such as violaxanthin, zeaxanthin,  $\beta$ -cryptoxanthin, and  $\beta$ -carotene, which reflect a more yellow-orange color (5,7,8).

Sometimes, color quality of red pepper products deteriorate during processing and storage resulting in decrease of market value. Usually, red pepper pods are dried using a natural solar drying or hot air drying methods and stored for several months, then ground to powder or flake forms before consumption. The color deterioration of dehydrated pepper products during drying and storage is attributed to the carotenoid destruction (9). Once the pods have been dehydrated and ground, the stability of the carotenoid pigments decreased (10-12). The stability of the pigments depends on cultivars, water activity, carotenoids level, and the endogenous antioxidants and enzyme activity (8).

Non-enzymatic browning is recognized as another dominant factor for affecting degradation of color quality of red pepper products (13,14). Ramakrishnan and Francis (13) showed that a major color change in heated paprika was due to the increase in the content of browning compounds. A non-enzymatic browning, Maillard reaction, is expected to occur in the dried red pepper since it contains appreciable amounts of reducing sugars and amino acids. Lee *et al.* (14) reported that browning reaction in dried red pepper products followed zero order reaction and the reaction rate was strongly affected by water activity and temperature.

To minimize undesirable color changes of dried red pepper products, it is necessary to have a good understanding of

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the main cause and degree of color change during processing and storage so that an effective packaging and storage system can be designed (14). Moisture content and storage temperature are recognized as 2 most important factors affecting the color quality of dried red pepper powder during handling and storage (3,9,15). Few research works have been published on the effect of water activity ( $A_w$ ) and temperature on the color change of red pepper, especially any approach based on moisture sorption isotherms has not been published yet.

The main objectives of this study were to determine the moisture sorption characteristics, and to investigate the effect of water activity and temperature on the color change of red pepper powder.

## Materials and Methods

**Materials** Freshly harvested and dried Asian pod type red peppers (*Capsicum annuum* L.; 11-13 g pod weight, 12-14 cm pod length, 2.6-3.2 cm pod diameter, pungency levels of 900-1,300 Scoville heat units) were obtained from Yeongyang Red Pepper Distribution Co. (Gyeongbuk, Korea). After de-stemming, only the pericarp was ground by using a rolling mill (Kyongchang Machinery, Seoul, Korea) to obtain red pepper powder used for experimental sample. The pepper powder sample was composed mostly of medium sized powder (58.82% with average particle diameters of 0.3325-0.6875 mm), followed by fine powder (35.59% with average particle diameter of 0.1475 mm), and large particles (5.59% with average particle diameter of 1.242 mm). The mass mean diameter of the pepper powder was 0.424 mm with the bulk density of  $0.49 \pm 0.02$  g/mL. Proximate compositions of the pepper powder sample were  $10.96 \pm 0.17$ ,  $10.85 \pm 0.26$ ,  $3.47 \pm 0.21$ , and  $8.30 \pm 0.18$  g/100 g powder for moisture, crude protein, crude fat, and ash, respectively.

**Moisture content** Moisture content of pepper samples was determined by drying at  $60^\circ\text{C}$  in a vacuum oven for 24 hr.

**Moisture sorption isotherms** Adsorption isotherms for the red pepper samples were determined using the static gravimetric method (16). Eight saturated salt solutions (LiCl,  $\text{K}_2\text{C}_2\text{H}_3\text{O}_2$ ,  $\text{MgCl}_2$ ,  $\text{K}_2\text{CO}_3$ ,  $\text{Mg}(\text{NO}_3)_2$ , NaCl, KCl, and  $\text{KNO}_3$ ) were used to maintain constant water activities ranging from 0.11 to 0.96 at 3 temperatures, 25, 40, and  $50^\circ\text{C}$  (17). About 3 g of red pepper samples weighed in aluminum dishes were placed in each of 8 hygrometers (sealed glass bottle) at different constant  $A_w$  established using the saturated salt solutions, and the equilibrium moisture content was measured after 20 days at the constant

temperature. Preliminary experiment indicated that it took about 15-20 days for the pepper samples to attain the equilibrium moisture content at each condition. Three replications were made at each  $A_w$ , and average values of moisture content were used for constructing the isotherm curves. The moisture content of each sample was based on the dry basis (g water/g dry solids), which was determined after each adsorption experiment using the oven drying method at  $105^\circ\text{C}$  for 24 hr. Weight of the pepper samples was determined using a digital balance (MC1 Analytic 210S; Satorius AG, Göttingen, Germany) with 0.1 mg accuracy.

The monolayer moisture contents of the pepper samples were determined using the Guggenheim-Anderson-de Boer (GAB) model:

$$m = \frac{m_o(CkA_w)}{(1-kA_w)(1-kA_w+CkA_w)} \quad (1)$$

where,  $m$  is the equilibrium moisture content (on dry weight basis) at  $A_w$ ;  $m_o$  is the moisture content of the monolayer corresponding to formation of a monomolecular layer on the internal surface of the pepper samples;  $C$  is the Guggenheim constant; and  $k$  is a factor corresponding properties of the multilayer molecules with respect to the bulk liquid. The parameters of the GAB model were estimated by a non-linear regression method employing the Marquardt-Levenberg algorithm using the Solver function of Excel<sup>®</sup> (18). The goodness of the fitting of the model to the experimental equilibrium moisture content and  $A_w$  data was evaluated on the basis of root mean square error ( $E_{RMS}$ ) as follows (19):

$$E_{RMS} = \left[ \frac{1}{N} \sum_{i=1}^N (m_{obs} - m_{pred})^2 \right]^{1/2} \quad (2)$$

where,  $m_{obs}$  and  $m_{pred}$  are the observed moisture content at any  $A_w$  and the corresponding predicted moisture content calculated using the GAB equation with the best fitted parameters, respectively, and  $N$  is the number of observations.

In addition, samples for examining the color changes caused by  $A_w$  and temperature were prepared separately by the same procedure for determining isotherms except for initial higher amount of pepper powder (about 30 g).

**Color measurement** Color change in the pepper powder samples was evaluated by measuring non-enzymatic browning, the ASTA values, and the Hunter- $L$ ,  $a$ ,  $b$  color values of the pepper powder sample. Non-enzymatic browning value was measured by the method of Lee *et al.* (14). Pepper powder sample (0.1 g) was suspended in 50 mL of distilled water, and water-soluble pigments were extracted at  $30^\circ\text{C}$  for 2 hr with intermittent stirring and then filtered through filter paper (No. 2; Toyo Roshi Kaisha,

Ltd., Tokyo, Japan). The absorbance of the filtrate was measured using a spectrophotometer (V-560; Jasco) at 420 nm against a blank of distilled water. The absorbance was converted to a dry weight of 0.1 g by compensating for the moisture content of the samples.

The ASTA color value was measured using the ASTA analytical method 20.1 (20). Pepper powder sample (70–100 mg) was added to 100 mL of acetone, and the mixture was stored at 0°C for 4 hr with intermittent stirring. The absorbance of an aliquot of the transparent extract was measured at 460 nm using a UV/VIS spectrophotometer (V-560; Jasco, Tokyo, Japan). ASTA color value was calculated as follows (2):

$$\text{ASTA color value} = A \times 16.4 \times C / w \quad (3)$$

where, A is absorbance of the extract, C is the correction factor of the spectrophotometer, which was calculated by dividing the theoretical absorbance by the real absorbance of standard color solution [0.001 M K<sub>2</sub>SO<sub>4</sub> and 0.09 M (NH<sub>4</sub>)<sub>2</sub>Co(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O in 1.8 M H<sub>2</sub>SO<sub>4</sub>] at 460 nm, and w is the sample weight (g) in dry basis.

The tristimulus Hunter L, a, and b-values of pepper powder sample were measured by reflectance using a chroma meter (CR-400; Konica-Minolta, Tokyo, Japan). The instrument was calibrated with a standard white tile (L=97.02, a=0.08, b=1.75) before measurements. Five readings at least were made separately from 3 different spots at sample cases (transparent mini petri dishes made of polypropylene) containing red pepper powder and the mean values were reported. Color functions such as total color difference (ΔE), hue angle (h), and chroma values were calculated from the Hunter L, a, and b values according to the following formulas:

$$\Delta E = [(L_o - L)^2 + (a_o - a)^2 + (b_o - b)^2]^{1/2} \quad (4)$$

$$h = 1 / \tan(b/a) \quad (5)$$

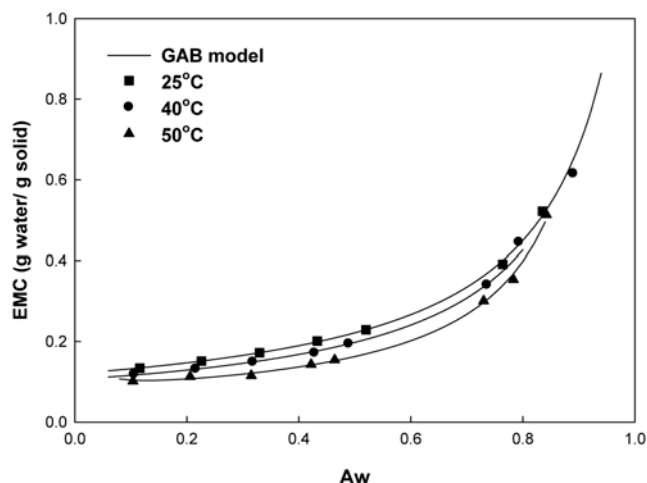
$$\text{Chroma} = (a^2 + b^2)^{1/2} \quad (6)$$

where, the L<sub>o</sub>, a<sub>o</sub>, and b<sub>o</sub> terms refer to the initial values for the untreated control pepper powder.

In addition, Hunter a×L and Hunter a/b values were used to test the color change of the red pepper powder samples (13).

## Results and Discussion

**Adsorption isotherms** The experimental results of equilibrium moisture content (EMC) of red pepper powder at each Aw for 3 different temperatures are presented in Fig. 1 with regression lines obtained by fitting the GAB model to the experimental data. The EMC of the red pepper powder samples varied in the range of 0.12 to 0.65 g water/g solid depending on Aw and temperature.



**Fig. 1.** Experimental and predicted (from GAB model) EMC and water activity (Aw) relationship for red pepper powder at different temperatures.

**Table 1.** GAB model parameters (*m<sub>o</sub>*, *k*, *C*) and the root mean square error (*E<sub>RMS</sub>*) of red pepper powder at different temperatures

Temp. (°C)	GAB constant			<i>E<sub>RMS</sub></i>
	<i>m<sub>o</sub></i> <sup>1)</sup>	<i>k</i>	<i>C</i>	
25	0.1218	0.92	39.80	0.0046
40	0.1078	0.98	101.87	0.0133
50	0.0912	1.02	413.58	0.0069

<sup>1)</sup>Unit, g water/g solid

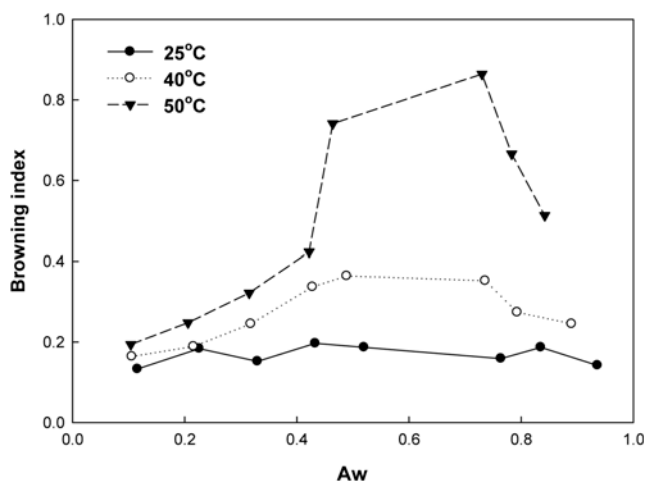
The isotherm curves show the characteristic S-shaped curve, typical of the sorption isotherms of many food materials (17). The EMC of red pepper powder increased linearly at low and intermediate Aw ranges and increased rapidly at high Aw region. Similar isotherm patterns were observed with dried red peppers (21), red bell pepper (22), and green and red peppers (23). Like other food materials, the EMC of the pepper powders decreased with increase in temperature at constant Aw indicating that the hygroscopicity of peppers is dependent on temperature. Such a change in hygroscopicity of pepper powders may be attributed to a change in the total number of active sites for water binding caused by temperature induced physicochemical changes in the product (24).

Figure 1 also shows that the GAB model fitted well to the experimental isotherm data of the red pepper powder. The GAB model parameters (*m<sub>o</sub>*, *k*, and *C*) for the red pepper powder samples at different temperatures were determined as shown in Table 1. The GAB model had high coefficient of determination (*r*<sup>2</sup>>0.975) and low root mean square errors (*E<sub>RMS</sub>*<0.01). The *E<sub>RMS</sub>* values are frequently used to test the goodness of fit of isotherms (24). Generally, good fitting of an isotherm is assumed when the *E<sub>RMS</sub>* value is less than 0.05 (19).

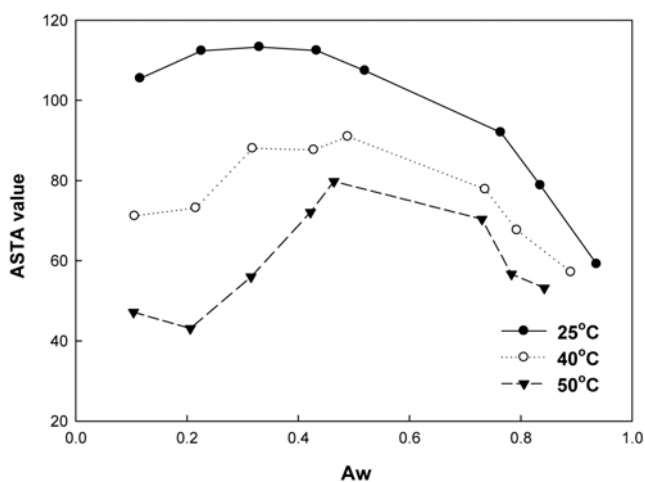
The monolayer moisture content ( $m_o$ ) which is the measure of the minimum moisture content covering hydrophilic sites on the red pepper powder was around the maximum value reported for food materials (25). The  $m_o$  values of red pepper powder were in good agreement with those observed in a various pepper products such as dried red pepper (21), red bell pepper (22), red pepper (23), and green pepper (23). The  $m_o$  values of red pepper powder decreased from 0.1218 to 0.0912 g water/g solid with increasing temperature from 25 to 50°C, respectively. Such decrease in  $m_o$  with temperature has been frequently observed in the isotherm tests with various food or biopolymer materials (26). Generally, the  $m_o$  values are recognized as criteria for achieving safe storage with minimum quality loss for a long time (24).

**Apparent color** Red pepper powder samples collected after isotherm tests indicated distinctive color changes from bright red to brownish and tarnished red depending on storage temperature and  $A_w$ . At 25°C, the pepper sample maintained brilliant red color at low  $A_w$  ranges up to 0.43, and changed into dark red with increasing darkness as  $A_w$  increased. At 40°C, the pepper sample showed bright red below  $A_w$  of 0.22 and it became darker with increase in  $A_w$ . However, it still maintained its red color up to  $A_w$  of 0.43 and turned dark red at  $A_w$  of 0.49, then became blackish red over  $A_w$  of 0.74. Much of red color was faded even at low  $A_w$  at 50°C. At this temperature, the pepper sample exhibited brownish red below  $A_w$  of 0.21, brown at 0.32, dark brown up to 0.46, then became almost black at higher  $A_w$  ranges. Such color changes of red pepper powder samples observed at temperature and  $A_w$  ranges are mainly attributed to not only destruction of carotenoid pigments but also development of browning compounds (1,2,14).

**Non-enzymatic browning** The relationship between browning index (BI) and water activity of pepper powder samples at different temperatures is shown in Fig. 2. Non-enzymatic browning of red pepper powder was not significantly influenced by the  $A_w$  at low temperature (25°C). However, browning increased as the temperature increased, especially at medium  $A_w$  ranges of 0.4–0.7. Lee and Park (27) and Lee *et al.* (14) also reported that the browning rate of red pepper was accelerated at higher temperature and moisture content. Lee *et al.* (14) determined the rate of non-enzymatic browning of red pepper using a zero-order reaction kinetics and found the reaction rate was higher at the medium range of  $A_w$  (@ 0.5–0.7), which is in accordance with the present result. It has been frequently observed that a major color change in red peppers during drying and storage was attributable to the non-enzymatic browning of red peppers due to their



**Fig. 2.** Effect of water activity ( $A_w$ ) and temperature on browning index of red pepper powder.



**Fig. 3.** Effect of water activity ( $A_w$ ) and temperature on ASTA color values of red pepper powder.

high contents of reducing sugars and amino acids (9,14, 28). Topuz *et al.* (4) tested the effect of drying method and storage on color characteristics and concluded that surface color degradation of paprika was more related to browning reaction than to carotenoids degradation.

**ASTA color values** The color quality of paprika is usually assessed by its oil extractable color, measured in ASTA units, which is directly related to the pigment content of red pepper or paprika. The ASTA values of red pepper powder determined at different temperatures and  $A_w$  values are shown in Fig. 3. The ASTA color value of the initial red pepper sample was  $118.4 \pm 0.1$ , which is in good agreement with that of red pepper (ASTA color value of 107–114) dried by hot air drying methods (2). ASTA values of the red pepper powder were greatly affected by both temperature and  $A_w$  of the product. As the temperature increased from

25 to 50°C, ASTA values of the red pepper powder decreased profoundly from 118.4 down to 43.1 depending on  $A_w$ , which indicates the importance of low storage temperature in keeping the color quality of the pepper powder. The ASTA values of red pepper powder were higher at a medium range of  $A_w$  (@ 0.4-0.5) at all temperatures tested. Above this  $A_w$  range (more than 0.5), ASTA color values decreased rapidly with increase in  $A_w$ , which is consistent with the result of non-enzymatic browning which increased above  $A_w$  of 0.5 (Fig. 2). At both extremes of  $A_w$  ranges, ASTA color values decreased significantly except a low  $A_w$  range of 25°C. Kim *et al.* (2) tested ASTA color values of red pepper powder sealed in a polyethylene/nylon laminated bag and stored at 0 and 22°C, and found that 72-85% of the color was retained after 6 months. This supports the present result of higher amount of color retained, i.e., higher ASTA color values, at lower temperature of storage.

The ASTA color value of red pepper is mainly attributed to the carotenoids such as capxanthin, capsorubin, and  $\beta$ -carotene (1). Carotenoids in some model foods and vegetables were reported to be more stable at an intermediate water activity range (9,29), which agrees well with the present result. Though ASTA provides values for the total coloring power of red pepper extract, it does not provide the levels of color saturation or the hue of red pepper (6).

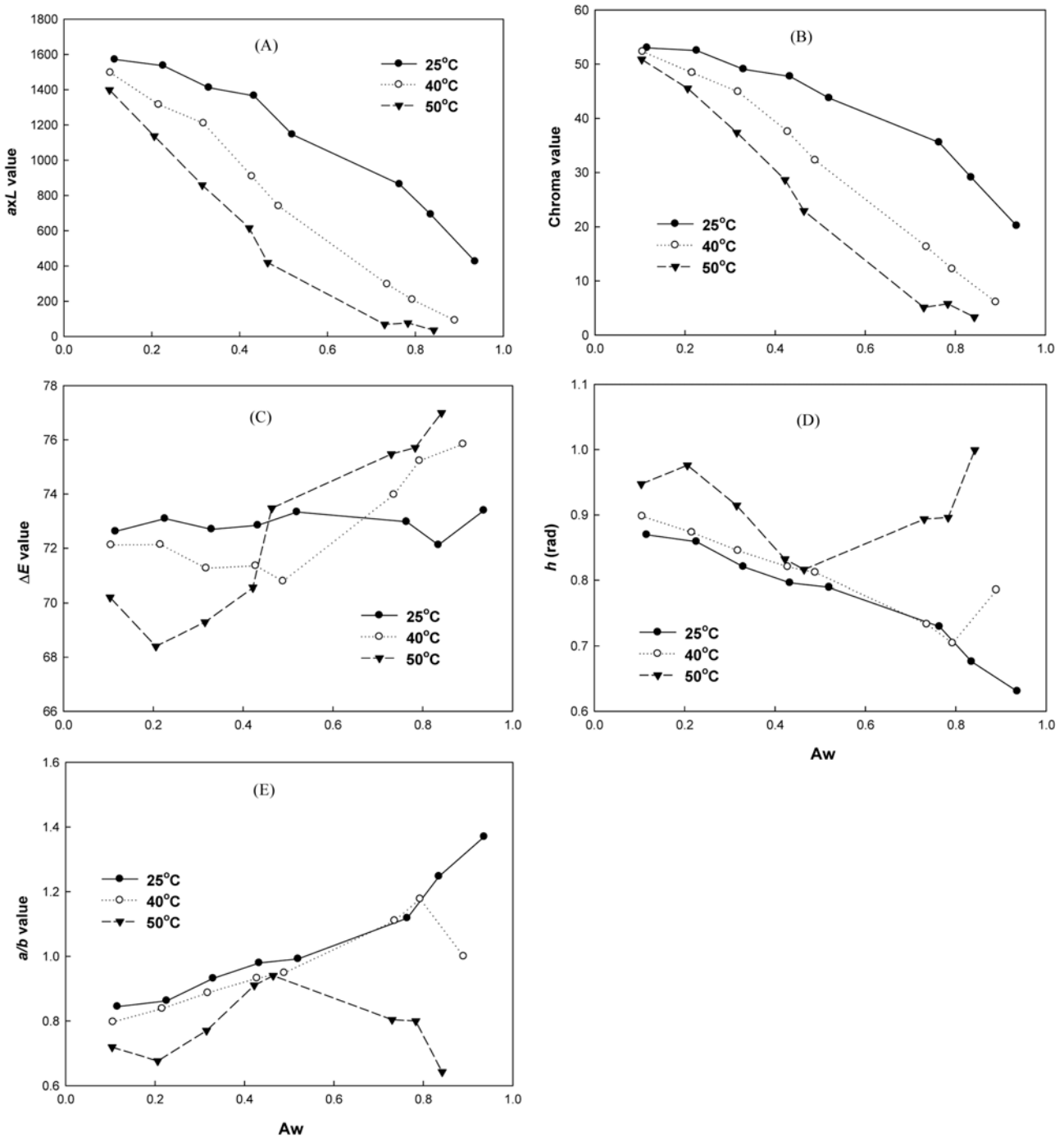
**Surface color** The apparent color of red pepper is featured by a mix of yellowness and redness due to the presence of carotenoids. These color components are represented by Hunter  $a$  and  $b$  color coordinates. Since any change in Hunter  $a$  and  $b$  values is accompanied by a simultaneous change in the  $L$  value (30), Hunter  $L$ ,  $a$ , and  $b$  values are usually used to define color characteristics of various peppers during drying and storage (2,4,14,15,31-33). The result of Hunter  $L$ ,  $a$ , and  $b$  values of red pepper powder determined at different temperature and  $A_w$  values are shown in Table 2. Hunter  $a$  values, which indicate redness of red pepper powder, decreased with increase in  $A_w$  at all temperatures tested. They also show significant effect of temperature, i.e., the lower the temperature, the higher the Hunter  $a$  value retained. Ramakrishnan and Francis (13) demonstrated the linear relationship between carotenoid content and Hunter  $a$  value of paprika. Hunter  $b$  values, which indicate yellowness of red pepper powder, showed a similar pattern of decrease with increase in  $A_w$  and temperature. Hunter  $L$  values, which are affected by both Hunter  $a$  and  $b$  values indicating lightness of red pepper powder, also showed a change against  $A_w$  and temperature. Hunter  $L$  value has been used as an indicator of non-enzymatic browning of red pepper (13). To examine the effect of temperature and  $A_w$  on color change of red pepper powder, combinations of the chromatic coordinates

**Table 2. Hunter  $L$ ,  $a$ ,  $b$  values of red pepper powder at different water activities ( $A_w$ ) and temperatures<sup>1)</sup>**

Temp. (°C)	$A_w$	Hunter- $L$	Hunter- $a$	Hunter- $b$
25	0.116	44.8±0.5	35.1±0.2	40.0±0.8
	0.226	44.3±0.3	35.3±0.3	40.0±0.2
	0.330	42.3±0.7	34.7±0.3	37.7±0.7
	0.433	39.2±1.8	33.0±2.0	33.8±2.2
	0.520	38.7±0.2	32.0±0.4	32.4±0.5
	0.764	34.0±0.3	27.5±0.6	24.8±0.4
	0.835	28.9±0.3	21.8±0.3	15.8±0.4
40	0.106	45.7±0.3	33.8±0.8	41.0±0.2
	0.216	43.3±0.3	33.4±0.5	37.9±0.2
	0.318	37.8±0.3	29.0±0.7	30.5±1.1
	0.428	34.1±0.4	24.7±0.7	24.7±0.8
	0.489	33.0±0.6	22.6±0.9	22.7±0.5
	0.736	25.2±0.1	11.5±0.6	9.9±0.7
	0.793	24.9±0.3	10.9±0.1	8.9±0.3
0.890	24.0±0.3	9.2±0.4	7.0±0.2	
50	0.104	48.0±0.8	29.9±0.3	42.5±0.7
	0.206	41.3±1.5	25.3±0.5	34.8±0.5
	0.315	35.6±0.5	15.9±0.2	23.9±0.3
	0.422	26.3±0.4	12.5±0.4	12.8±0.2
	0.464	31.2±0.9	10.7±0.2	16.9±0.1
	0.730	21.9±0.3	3.0±0.2	3.8±0.1
	0.783	21.3±0.4	2.8±0.1	3.5±0.1
0.842	21.0±0.4	2.3±0.4	3.3±0.3	

<sup>1)</sup>Hunter- $L$ ,  $a$ ,  $b$  values of control pepper powder were 43.54±0.5, 31.8±0.3, and 37.7±0.5, respectively.

such as  $E$ , chroma, hue angle,  $a \times L$ , and  $a/b$  values were plotted against  $A_w$  at different temperatures as shown in Fig. 4. Ramakrishnan and Francis (13) showed that Hunter  $L$  values increased linearly with increase in browning compounds in paprika. Hunter  $a \times L$  values of red pepper powder (Fig. 4A) also exhibited a similar trend of change against  $A_w$  and temperature as Hunter  $a$  and  $L$  values. It is interesting to note that the changing pattern of  $a \times L$  values is more close to that of Hunter  $a$  values rather than that of Hunter  $L$  values. Ramakrishnan and Francis (13) demonstrated  $a \times L$  values could be used appropriately as an index of color degradation of paprika caused by heating. They characterized paprika color based on  $a \times L$  values such that paprika samples with  $a \times L$  value above 500 would be rated visually red; the values between 300 and 500 would be medium red and the values below 300 would be rated as dark. They also recognized paprika samples with  $a \times L$  values above 700 appeared brilliant red. In the present study,  $a \times L$  value of red pepper powder decreased from 1,380 for control sample to below 700 above medium  $A_w$  value of around 0.76, 0.48, and 0.38 at temperature of 25, 40, and 50°C, respectively. This also shows a compound effect of  $A_w$  and temperature on color degradation of red



**Fig. 4.** Effect of water activity ( $A_w$ ) and temperature on tristimulus Hunter color values and their color functions. (A)  $a \times L$  value, (B) chroma value, (C) total color difference ( $\Delta E$ ) value, (D) hue angle ( $h$ ) value, (E)  $a/b$  value

pepper powder indicating that storage at low temperature and moisture content is desirable to maintain the color quality of red pepper powder. Chroma values of red pepper powder, which indicate degree of saturation of color, also exhibited the very similar change against  $A_w$  and temperature as  $a \times L$  values (Fig. 4B). This indicates that chroma value can also be properly used as an index for color change of red pepper powder. Lee *et al.* (14) reported that chroma

value was highly correlated with browning during storage of red pepper powder. By the way, total color difference ( $\Delta E$ ) values of red pepper powder show a quite different pattern of change against  $A_w$  and temperature from the previous color indices (Fig. 4C). It is known that the difference in 0.15  $\Delta E$  units of food sample can be distinguished by a trained panel (13). It is noteworthy that  $\Delta E$  values changed slightly with increase in  $A_w$  at a low

temperature (25°C), however, they increased significantly after medium  $A_w$  value at both 40 and 50°C. This means color of red pepper powder changes more at higher temperature and  $A_w$  ranges. Hue angle ( $h$ ) of red pepper powder changed against  $A_w$  and temperature as shown in Fig. 4D. The  $h$  value is defined as a color wheel, with red-purple at an angle of 0, yellow at  $\pi/2$ , bluish-green at  $\pi$  and blue at  $3\pi/2$  rad. Generally,  $h$  values of red pepper powder decreased with increase in  $A_w$  except 50°C and they increased with increase in temperature. This indicates higher temperature of storage induce change in pepper color from red to brown. Hunter  $a/b$  values of red pepper powder, which represent a red/yellow color ratio, were determined to evaluate color change at different  $A_w$  and temperatures (Fig. 4E). Hunter  $a/b$  values of red pepper powder exhibited the reversed pattern of change observed in  $h$  values. Ramakrishnan and Francis (13) tested  $a/b$  values to represent the color change in paprika heated at different temperatures and found that this color value decreased with increase in heating time and temperature with high correlation with corresponding carotenoid losses and increases in browning compounds.

In conclusion, the change in color of red pepper powder was remarkably influenced by both temperature and  $A_w$ . As storage temperature and  $A_w$  increased, the color of red pepper powder faded out from brilliant red to dull brown and eventually to tarnish black. Therefore, it is desirable to store red pepper powder at a low temperature below 25°C and below medium ranges of  $A_w$  to keep the color quality. All the color parameters tested properly represented such color changes in red pepper powder. These parameters can be used appropriately to evaluate color degradation of red pepper products during drying and storage. They can also be used for practical purposes in controlling the color characteristics of the product.

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