

## Characteristics of Sucrose Thermal Degradation with High Temperature and High Pressure Treatment

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**Abstract** Thermal degradation characteristics of sucrose was investigated. A 20% sucrose solution was heated to temperatures of 110-150°C for 1-5 hr. Chromaticity, pH, organic acids, 5-hydroxymethylfurfural (HMF), free sugars, electron donating ability (EDA), and ascorbic acid equivalent antioxidant capacity (AEAC) of the heated sucrose solutions were evaluated. With increasing temperatures and times, the L-, a-, and b-values decreased; however, total color difference ( $\Delta E_{ab}$ ) increased. The pH and sucrose contents decreased, and fructose and glucose contents increased with increasing heating temperature and time. Organic acids, such as formic acid, lactic acid, and levulinic acid, and HMF contents increased with increasing heating temperatures and times. EDA (%) and the AEAC of the heated sucrose solutions increased with increasing heating temperature and time. The heated sucrose solution was more effective than unheated sucrose solution, having higher EDA (90 fold), and AEAC (13 fold).

**Keywords:** sucrose thermal degradation, electron donating ability (EDA), 5-hydroxymethylfurfural (HMF), heating temperature and time, organic acid

### Introduction

The thermal degradation of sucrose is an important reaction in the food industry, because it is responsible for important characteristics of the final food products, and it can influence the yield of white sugar obtainable in sugar manufacturing (1). Sucrose degradation occurs primarily by two reaction pathways: the Maillard reaction, which takes place in the presence of amino acids, and caramelization, which occurs when sucrose is heated to high temperature (2). The kinetics of the Maillard reaction has been widely studied in the food science field (3-5). Caramelization, on the other hand, is considered to be a simpler reaction pathway, and fewer studies have been dedicated to its kinetics (6).

The caramelization reaction is influenced by pH and sucrose concentration in the solution, and several authors have studied this (7-9). A first-order model is often used to describe the heat degradation of sucrose under such conditions (10). Little information is available on the kinetics of the thermal degradation of sucrose at high concentrations, excepting a study by Schoebel *et al.* (11).

Eggleston *et al.* (12) modeled the reaction assuming a pseudo-first-order reaction for concentrated sucrose thermal degradation in the presence of different salts. A lag phase was also observed by Eggleston (13), when canning juice was stored at room temperature, where sucrose degradation was due to the combined action of microbial, enzymatic, and chemical (acidic degradation) reactions. Haghghat *et al.* (14) found this type of behavior in the thermal degradation of sucrose in subcritical water (high pressure and high temperature conditions). An induction period was

also observed in the non-enzymatic browning of freeze-dried model systems containing sucrose (15,16).

Recent studies have shown that thermally processed foods, especially fruits and vegetables, have higher biological activity, because of chemical changes during heat treatment (17). Some studies have examined the chemical and physical properties of foods in response to high temperature and high pressure (HTHP) treatment. The polyphenol and flavonoid contents and antioxidant activity increase with HTHP treatment in foods such as *shiitake* mushrooms (18), garlic (19), onions (20,21), ginseng (22), and pears (23). To our knowledge, however, no study has examined changes in physicochemical characteristics and antioxidant activity in a sucrose model system, with varying temperature and time.

Thus, our objective was to investigate the thermal degradation characteristics of a sucrose model system at various temperatures and times, and to analyze the changes in antioxidant activity, chromaticity, pH, organic acids, 5-hydroxymethylfurfural (HMF), and sucrose content.

### Materials and Methods

**Materials** Sucrose, 1,1-diphenyl-2-picrylhydrazyl (DPPH), 2,2-azino-bis-3-ethylbenzothiazoline-6-sulfonic acid (ABTS), potassium persulfate, acetic acid, citric acid, formic acid, lactic acid, and levulinic acid were purchased from Sigma-Aldrich (St. Louis, MO, USA). 5-Hydroxymethylfurfural (HMF) was purchased from Wako Pure Chemical Inc. (Osaka, Japan). Water, acetonitrile, and methanol were purchased from J.T. Baker (San Francisco, CA, USA). All other reagents were of analytical grade.

**Sample treatment** A 20%(w/w) sucrose solution was prepared by weighing sucrose and adding distilled water in the desired proportion. Sucrose solution was placed in a

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sample bottle and sealed tightly. Sample bottles were heated using high-pressure steam, generated by a temperature-controlling apparatus (Jisico, Seoul, Korea). Samples were heated to temperature of 110, 120, 130, 140, or 150°C for 1, 2, 3, 4, or 5 hr (19-25). Heated samples were centrifuged (1,800×g, 10 min). The supernatant was filtered through a 0.45- $\mu$ m syringe filter (Millipore, Billerica, MA, USA). Extracts were kept at -20°C until analysis.

**Color measurement** Samples were poured into a clear glass petri dish, and color parameters were determined using a tristimulus colorimeter (Chroma Meter CR300; Konica Minolta Holdings, Inc., Tokyo, Japan). Results were calculated by the equipment based on the Hunter Lab color scale. Color changes in a sucrose solution are commonly evaluated through changes in the total color difference ( $\Delta E_{ab}$ ) parameter, which evaluates the overall color difference of a heated sample compared to an untreated sample:

$$\Delta E_{ab} = [(L - L_0)^2 + (a - a_0)^2 + (b - b_0)^2]^{1/2}$$

where L-value ranges from 0 (black) to 100 (white), a-value indicates degree of greenness (for negative a-values) and degree of redness (for positive a-values), b-value also ranges from negative to positive values indicating, respectively, degree of blueness to yellowness.

**pH and organic acid contents** A pH meter (Model 320; Thermo Orion, Beverly, MA, USA) was used for the determination of pH values. Organic acid contents were measured according to a modification of the method of Šturm *et al.* (26). The separation of organic acids was performed with an analytical high performance liquid chromatograph (HPLC; Thermo Separation Products, San Jose, CA, USA), using an Aminex Ion Exclusion HPX-87H (7.8×300 mm, Bio-Rad Laboratories, Hercules, CA, USA) column with a guard column (Aminex Cation-H guard column; Bio-Rad). Elution was carried out at a solvent flow rate of 0.6 mL/min, isocratically, with sulfuric acid (0.008 N) as the mobile phase. Detection was performed with a ultraviolet (UV) detector set at 215 nm. All samples were analyzed in triplicate.

**Analysis of free sugars** The free sugar content was measured according to a modification of the method of Woo *et al.* (20), using fructose, glucose, and sucrose as standards for calibration curves. Samples were filtered through a 0.45- $\mu$ m syringe filter (Millipore) and analyzed by HPLC (Waters 2695; Waters, New Castle, DE, USA). The analytical column was for carbohydrates (4.6×150 mm, Waters) and the mobile phase was water-acetonitrile (25:75, v/v) at a flow rate of 1 mL/min. The injection volume was 20  $\mu$ L, and the detector was an evaporative light scattering detector (Waters 2420). All samples were analyzed in triplicate.

**Analysis of HMF contents** HMF contents were measured according to a modification of the method of Kwon *et al.* (19). Samples were filtered through a 0.45- $\mu$ m syringe filter (Millipore) and analyzed by HPLC (Thermo Separation Products). The analytical column was an LC-18 (4.6×250

mm), obtained from Phenomenex (Torrance, CA, USA). Water-acetonitrile (80:20, v/v) was used at a flow rate of 0.8 mL/min. The injection volume was 20  $\mu$ L, and the UV detector was set at 280 nm. The standard used was HMF, and all samples were analyzed in triplicate.

**Antioxidant activity using the DPPH assay** The scavenging activity of samples for the DPPH radical was measured according to the method of Tepe *et al.* (27), with some modifications. An aliquot of 0.8 mL of 0.2 mM DPPH methanolic solution was mixed with 0.2 mL of sample. The mixture was shaken vigorously and left to stand for 30 min under low light. The absorbance was measured at 520 nm. The DPPH radical scavenging activity (%) was calculated as:

$$\text{DPPH radical scavenging activity (\%)} = (1 - A_{\text{sample}}/A_{\text{control}}) \times 100$$

where  $A_{\text{sample}}$  is the absorbance in the presence of sample and  $A_{\text{control}}$  is the absorbance in the absence of sample. All samples were analyzed in triplicate.

**ABTS cation radical scavenging activity** The scavenging activity of the extracts for the ABTS cation radical was measured according to the method of Re *et al.* (28), with some modifications. The ABTS cation radical was generated by adding 7 mM ABTS to 2.45 mM potassium persulfate solution, and leaving the mixture to stand overnight in the dark at room temperature. The ABTS cation radical solution was diluted with distilled water to obtain an absorbance of 1.4-1.5 at 735 nm (molar extinction coefficient,  $\epsilon = 3.6 \times 10^4$ /mol/cm). Diluted ABTS cation radical solution (1 mL) was added to 50  $\mu$ L of extract, ascorbic acid standard solution, or distilled water. After 60 min, the absorbance was measured at 735 nm using a spectrophotometer (DU-650; Beckman, Fullerton, CA, USA). The ABTS cation radical scavenging activity was expressed in terms of ascorbic acid equivalent antioxidant capacity (AEAC), as mg of ascorbic acid equivalents/g sample (mg AA eq/g sample) (29).

**Statistical analysis** All data are expressed as mean  $\pm$  standard deviation (SD). The significance of differences among treatment means was determined by one-way analysis of variance (ANOVA) using SAS (version 9.1, SAS Institute, Cary, NC, USA) with a significance level of 0.05.

## Results and Discussion

**Color** The changes in chromaticity of the 20% sucrose solution under different heating conditions are shown in Table 1. Significant differences in the total color difference ( $\Delta E_{ab}$ ) of heated sucrose solution were observed under various heating conditions. Heating temperature caused more pronounced effects than heating time ( $p < 0.001$ ; Table 3). The  $\Delta E_{ab}$  values increased significantly with increased heating temperature (110 to 150°C) and time (1 to 5 hr;  $p < 0.001$ ). The L-, a-, and b-values of the unheated sucrose solution were 40.67, 0.42, and -1.48, respectively. With increasing heating temperature and time, the L-value decreased. The a-value decreased to -0.57 at 130°C for 1 hr and increased after that. The b-value increased to

**Table 1. The chromaticity of 20% sucrose solution treated with high temperature and high pressure**

Temp. (°C)	Time (hr)	L	a	b	$\Delta E_{ab}^{1)}$
	Control	40.67±0.75 <sup>a2)</sup>	0.42±0.03 <sup>jk1</sup>	-1.48±0.02 <sup>u</sup>	-
110	1	40.22±0.12 <sup>ab</sup>	0.31±0.06 <sup>jk1</sup>	-1.28±0.08 <sup>tu</sup>	0.65±0.61 <sup>k</sup>
	2	40.42±1.03 <sup>ab</sup>	0.34±0.02 <sup>jk1</sup>	-1.20±0.09 <sup>t</sup>	1.21±0.44 <sup>jk</sup>
	3	40.47±0.11 <sup>ab</sup>	0.23±0.02 <sup>jklm</sup>	-0.76±0.02 <sup>s</sup>	0.95±0.31 <sup>jk</sup>
	4	40.60±0.69 <sup>a</sup>	-0.05±0.04 <sup>lmn</sup>	0.03±0.03 <sup>r</sup>	1.60±0.05 <sup>jk</sup>
	5	40.75±0.35 <sup>a</sup>	-0.19±0.04 <sup>lmno</sup>	0.61±0.01 <sup>q</sup>	2.21±0.05 <sup>j</sup>
120	1	40.55±0.57 <sup>ab</sup>	0.17±0.03 <sup>klm</sup>	-0.76±0.04 <sup>s</sup>	1.24±0.48 <sup>jk</sup>
	2	40.39±0.12 <sup>ab</sup>	-0.76±0.04 <sup>o</sup>	2.79±0.08 <sup>k</sup>	4.49±0.16 <sup>i</sup>
	3	39.60±0.61 <sup>b</sup>	-1.60±0.08 <sup>p</sup>	6.99±0.25 <sup>g</sup>	8.78±0.22 <sup>h</sup>
	4	38.04±0.19 <sup>c</sup>	-1.41±0.03 <sup>p</sup>	10.48±0.24 <sup>c</sup>	12.39±0.38 <sup>f</sup>
	5	36.34±0.12 <sup>d</sup>	-0.41±0.03 <sup>mno</sup>	11.95±0.25 <sup>b</sup>	14.15±0.07 <sup>e</sup>
130	1	37.46±0.28 <sup>c</sup>	-0.57±0.05 <sup>no</sup>	8.94±0.07 <sup>e</sup>	11.07±0.30 <sup>g</sup>
	2	32.77±0.24 <sup>e</sup>	1.31±0.07 <sup>hi</sup>	13.61±0.13 <sup>a</sup>	17.07±0.45 <sup>d</sup>
	3	26.51±0.43 <sup>f</sup>	4.89±0.03 <sup>b</sup>	9.58±0.12 <sup>d</sup>	18.51±0.38 <sup>c</sup>
	4	22.07±0.41 <sup>gh</sup>	4.03±0.09 <sup>c</sup>	3.27±0.06 <sup>j</sup>	19.53±0.45 <sup>bc</sup>
	5	20.64±0.41 <sup>j</sup>	1.45±0.04 <sup>ghi</sup>	2.29±0.04 <sup>l</sup>	20.40±0.52 <sup>ab</sup>
140	1	26.11±0.33 <sup>f</sup>	5.56±0.01 <sup>a</sup>	8.15±0.09 <sup>f</sup>	18.20±0.80 <sup>cd</sup>
	2	22.76±0.55 <sup>g</sup>	2.94±0.06 <sup>de</sup>	3.63±0.24 <sup>i</sup>	18.79±0.73 <sup>c</sup>
	3	22.56±0.60 <sup>gh</sup>	3.10±0.05 <sup>d</sup>	4.15±0.07 <sup>h</sup>	19.15±0.54 <sup>bc</sup>
	4	22.51±1.02 <sup>gh</sup>	1.51±0.14 <sup>gh</sup>	1.99±0.31 <sup>m</sup>	18.52±0.61 <sup>c</sup>
	5	21.64±0.28 <sup>hi</sup>	0.83±0.04 <sup>hijk</sup>	1.70±0.03 <sup>n</sup>	19.29±0.78 <sup>bc</sup>
150	1	22.27±0.63 <sup>eh</sup>	2.30±0.06 <sup>ef</sup>	3.41±0.07 <sup>ji</sup>	19.13±0.89 <sup>bc</sup>
	2	21.93±0.48 <sup>eh</sup>	2.30±0.09 <sup>ef</sup>	3.00±0.27 <sup>k</sup>	19.35±0.92 <sup>bc</sup>
	3	20.90±0.26 <sup>ji</sup>	2.08±0.01 <sup>fg</sup>	2.42±0.06 <sup>l</sup>	20.21±0.90 <sup>ab</sup>
	4	19.48±0.65 <sup>k</sup>	0.95±0.09 <sup>hij</sup>	1.15±0.17 <sup>o</sup>	21.35±0.87 <sup>a</sup>
	5	19.64±0.55 <sup>k</sup>	0.78±0.08 <sup>ijk</sup>	0.85±0.10 <sup>p</sup>	21.16±0.70 <sup>a</sup>

<sup>1)</sup> $\Delta E_{ab}$ : total color difference= $[(L-L_0)^2+(a-a_0)^2+(b-b_0)^2]^{1/2}$

<sup>2)</sup>Each value is mean±SD (n=3); Any means in the same column followed by the same letter are not significantly (p<0.05) different by Duncan's multiple-range test.

13.61 at 130°C for 2 hr and decreased after that. The  $\Delta E_{ab}$  value suddenly increased after 120°C for 3 hr, and the maximum value was 21.35 at 150°C for 4 hr. The solution became obviously darker with increased heating temperature and time (7,8).

**pH** The changes in pH of the 20% sucrose solution under various heating conditions are shown in Table 2. Significant differences were observed with heating (p<0.001; Table 3). The pH of the distilled water in which the sample was dissolved was about 5.00 and that of the unheated sucrose solution was 4.55. The pH became more acidic at all heating conditions and was between 2.34 and 4.38. At a given heating time, higher temperatures resulted in lower pH values. At a given heating temperature, longer times yielded lower pH values. The decrease in pH with heating can be attributed to increased production of organic acids, such as formic acid, lactic acid, and levulinic acid (30,31). Higher temperature and longer time seemed to promote the production of organic acids.

**Organic acid content** The changes in organic acid content of the 20% sucrose solution with heating are shown in Table 2. No organic acid was detected in the unheated

sucrose solution. Significant differences were observed with heating (p<0.001; Table 3). The contents of organic acids, such as formic acid, lactic acid, and levulinic acid, increased significantly with increasing heating temperature (110 to 150°C) and time (1 to 5 hr; p<0.001). Formic acid was not detected below 120°C for 4 hr. The content of formic acid was 0.18 mg/mL at 120°C for 5 hr and increased after that. The maximum content of formic acid was 2.35 mg/mL at 150°C for 4 hr. Lactic acid was not detected below 120°C for 3 hr. The content of lactic acid was 0.03 mg/mL at 120°C for 4 hr and increased after that. The maximum content of formic acid was 4.16 mg/mL at 150°C for 5 hr. Levulinic acid increased with increasing heating temperature and time. The maximum content of levulinic acid was 15.35 mg/mL at 150°C for 4 hr. Aida *et al.* (30) and Shaw *et al.* (32) reported strong relationships between organic acid content and heating conditions.

**Free sugar content** The changes in sucrose content of the 20% sucrose solution with heating conditions are shown in Fig. 1. Significant differences were observed (p<0.001; Table 3). The sucrose content decreased significantly with increased heating temperature (110 to 120°C) and time (1 to 5 hr; p<0.001). The sucrose content slowly decreased at

**Table 2. The pH and organic acid contents of 20% sucrose solution treated with high temperature and high pressure**

Variables		pH	Concentration (mg/mL)		
Temp. (°C)	Time (hr)		Formic acid	Lactic acid	Levulinic acid
	Control	4.55±0.04 <sup>a1)</sup>	-	-	-
110	1	4.38±0.02 <sup>b</sup>	-	-	0.204±0.004 <sup>j</sup>
	2	4.20±0.02 <sup>c</sup>	-	-	0.312±0.011 <sup>hij</sup>
	3	4.07±0.03 <sup>d</sup>	-	-	0.327±0.012 <sup>hij</sup>
	4	3.99±0.02 <sup>e</sup>	-	-	0.349±0.009 <sup>hij</sup>
	5	3.71±0.03 <sup>f</sup>	-	-	0.376±0.007 <sup>hij</sup>
120	1	4.03±0.02 <sup>e</sup>	-	-	0.244±0.008 <sup>ij</sup>
	2	3.60±0.03 <sup>h</sup>	-	-	0.316±0.009
	3	3.37±0.01 <sup>i</sup>	-	-	0.348±0.012 <sup>hij</sup>
	4	3.26±0.02 <sup>k</sup>	-	0.028±0.002 <sup>g</sup>	0.359±0.015 <sup>hij</sup>
	5	3.09±0.01 <sup>m</sup>	0.180±0.002 <sup>l</sup>	0.098±0.004 <sup>fg</sup>	0.417±0.017 <sup>hij</sup>
130	1	3.32±0.02 <sup>j</sup>	0.245±0.008 <sup>kl</sup>	-	0.282±0.011 <sup>ghij</sup>
	2	3.05±0.02 <sup>n</sup>	0.441±0.011 <sup>hi</sup>	0.024±0.001 <sup>g</sup>	0.689±0.024 <sup>ij</sup>
	3	2.82±0.07 <sup>s</sup>	0.602±0.015 <sup>g</sup>	0.044±0.001 <sup>fg</sup>	0.763±0.031 <sup>ghij</sup>
	4	2.79±0.01 <sup>s</sup>	0.804±0.021 <sup>f</sup>	0.072±0.002 <sup>fg</sup>	1.071±0.036 <sup>fgh</sup>
	5	2.74±0.01 <sup>t</sup>	0.914±0.031 <sup>e</sup>	0.172±0.005 <sup>f</sup>	2.527±0.052 <sup>f</sup>
140	1	3.20±0.01 <sup>l</sup>	0.307±0.012 <sup>jk</sup>	-	0.347±0.012 <sup>d</sup>
	2	3.00±0.01 <sup>o</sup>	0.512±0.024 <sup>h</sup>	0.035±0.002 <sup>fg</sup>	0.377±0.018 <sup>hij</sup>
	3	2.96±0.01 <sup>p</sup>	0.811±0.028 <sup>f</sup>	0.378±0.002 <sup>e</sup>	0.499±0.021 <sup>ghij</sup>
	4	2.91±0.02 <sup>q</sup>	0.870±0.031 <sup>ef</sup>	0.444±0.001 <sup>ed</sup>	1.471±0.037 <sup>e</sup>
	5	2.86±0.01 <sup>r</sup>	1.319±0.035 <sup>c</sup>	0.483±0.002 <sup>ed</sup>	3.133±0.064 <sup>c</sup>
150	1	2.70±0.02 <sup>u</sup>	0.375±0.016 <sup>ij</sup>	0.046±0.001 <sup>fg</sup>	0.835±0.021 <sup>f</sup>
	2	2.52±0.01 <sup>v</sup>	0.668±0.019 <sup>g</sup>	0.548±0.013 <sup>d</sup>	1.684±0.052 <sup>e</sup>
	3	2.43±0.02 <sup>w</sup>	1.076±0.028 <sup>d</sup>	2.682±0.052 <sup>c</sup>	11.745±0.128 <sup>b</sup>
	4	2.39±0.02 <sup>x</sup>	2.346±0.062 <sup>a</sup>	2.945±0.056 <sup>b</sup>	15.348±0.243 <sup>a</sup>
	5	2.34±0.01 <sup>y</sup>	1.637±0.042 <sup>b</sup>	4.161±0.043 <sup>a</sup>	11.954±0.176 <sup>b</sup>

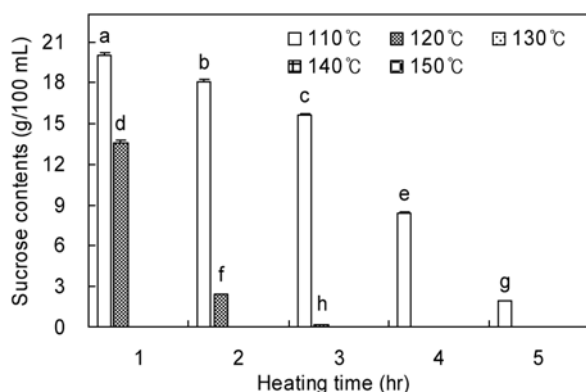
<sup>1)</sup>Each value is mean±SD ( $n=3$ ); Any means in the same column followed by the same letter are not significantly ( $p<0.05$ ) different by Duncan's multiple-range test.

**Table 3. Analysis of variance for pH, levulinic acid, total color difference ( $\Delta E_{ab}$ ), 5-hydroxymethylfurfural (5-HMF), sucrose content, electron donating ability (EDA), and ascorbic acid equivalent antioxidant capacity (AEAC) under various heating conditions**

	Variables <sup>1)</sup>	df	Sum of square	Mean of square	F-value
pH	X <sub>1</sub>	4	21.88	5.47	570.19**** <sup>2)</sup>
	X <sub>2</sub>	4	2.85	0.71	74.24***
Levulinic acid content	X <sub>1</sub>	4	693.78	173.45	28.36***
	X <sub>2</sub>	4	148.92	37.23	6.09***
$\Delta E_{ab}$	X <sub>1</sub>	4	3,955.76	988.94	232.31***
	X <sub>2</sub>	4	273.21	68.30	16.04***
5-HMF content	X <sub>1</sub>	4	1,152,023.81	288,005.95	350.32***
	X <sub>2</sub>	4	71,549.30	17,887.32	21.76***
Sucrose content	X <sub>1</sub>	4	1,835.39	458.85	41.31***
	X <sub>2</sub>	4	350.59	87.65	7.89***
EDA	X <sub>1</sub>	4	41,527.93	10,381.98	103.58***
	X <sub>2</sub>	4	5,175.98	1,294.00	12.91***
AEAC	X <sub>1</sub>	4	1,461.50	365.38	104.32***
	X <sub>2</sub>	4	257.85	64.46	18.40***

<sup>1)</sup>X<sub>1</sub>, heating temperature (°C); X<sub>2</sub>, heating time (hr).

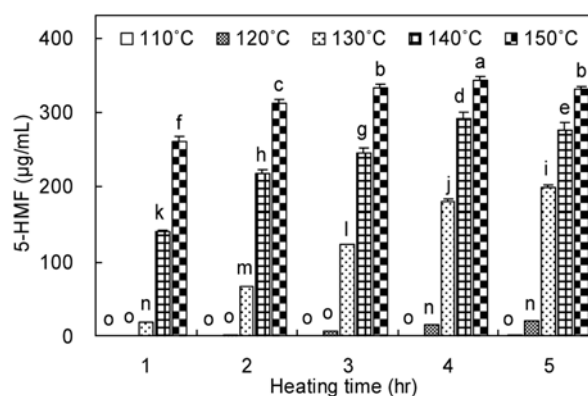
<sup>2)</sup>\*\*\* $p<0.001$ .



**Fig. 1. Sucrose contents of 20% sucrose solution treated with high temperature and high pressure.** (untreated sucrose solution: 20.16±0.11 g/100 mL).

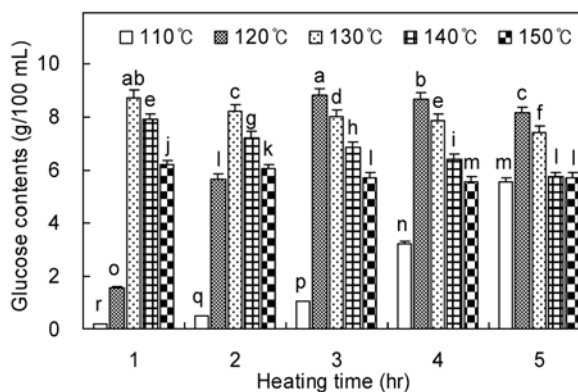
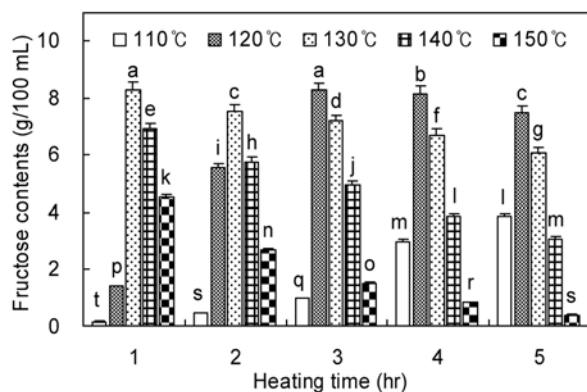
110°C, but abruptly decreased at 120°C with increasing heating time. The fructose and glucose contents under different heating conditions are shown in Fig. 2. Fructose and glucose contents slowly increased with heating until 120°C for 1 hr, but then abruptly increased after that. Fructose and glucose contents then decreased after 120°C for 5 hr and 130°C for 1 hr, respectively. The fructose and glucose contents of the heated sucrose solution at 130°C for 1 hr were 8.30 and 8.71 g/100 mL, respectively. Caramelization is the common name for a group of reactions that occurs when carbohydrates are exposed to high temperatures. They often occur during the preparation of traditional sucrose syrups and caramels, which are extensively used in confectionery and pastry products (6). Heat treatment of sucrose results in its degradation to fructose and glucose, and then heat degradation products, such as furfural, 5-methylfurfural, HMF, formic acid, lactic acid, and levulinic acid (33).

**HMF contents** The HMF content of the 20% sucrose solution under different heating conditions is shown in Fig. 3. HMF is a key furan derivative, readily derivable from renewable resources like carbohydrates, especially through the acid-catalyzed dehydration of fructose or fructose precursors. HMF is a suitable starting material for the

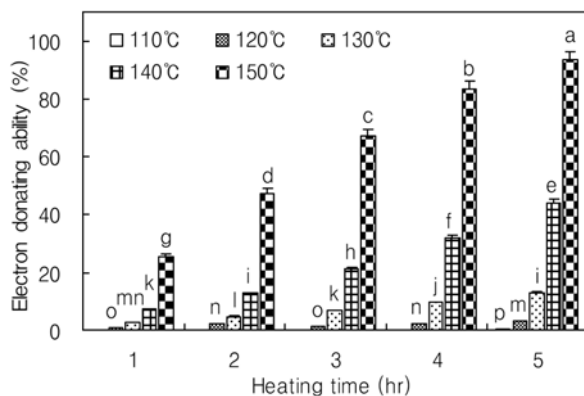


**Fig. 3. 5-Hydroxymethylfurfural (HMF) contents of 20% sucrose solution treated with high temperature and high pressure.** (untreated sucrose solution: 0.07±0.01 µg/mL).

preparation of further furanic monomers required for the preparation of non-petroleum-derived polymeric materials, such as polyesters, polyamides, and polyurethanes (34). Significant differences in the HMF content of the heated sucrose solution were observed with heating ( $p < 0.001$ ; Table 3). The HMF content of heated sucrose solution increased significantly with increasing heating temperature (110 to 150°C) and time (1 to 5 hr;  $p < 0.001$ ). The HMF content increased slowly below 130°C for 2 hr, and then suddenly increased after that. The HMF contents were 65.99 and 122.91 µg/mL at 130°C for 2 and 3 hr, respectively. The HMF content increased to 150°C for 4 hr, and decreased after that. The maximum HMF content was 342.44 µg/mL at 150°C for 4 hr. Very high levels of HMF have been reported in some food products, such as dried pears (3.5 g/kg), caramel products (9.5 g/kg) (35), instant coffee powder (6.2 g/kg), and coffee substitutes (13.9 g/kg) (36). HMF is one of the chief products of carbohydrate degradation in food, known as non-enzymatic browning. It has been demonstrated that the HMF level in a saccharidic foodstuff increases greatly during thermal treatment and its concentration follows a kinetic model of pseudo first-order (37). Quintas *et al.* (6) reported that after an initial phase of delay, sucrose content rapidly decreases and glucose and fructose start to form. Short time after, HMF is detected, followed



**Fig. 2. Fructose and glucose contents of 20% sucrose solution treated with high temperature and high pressure.** Fructose and glucose contents of untreated sucrose solution were 0.16±0.01 and 0.16±0.01 g/100 mL, respectively.

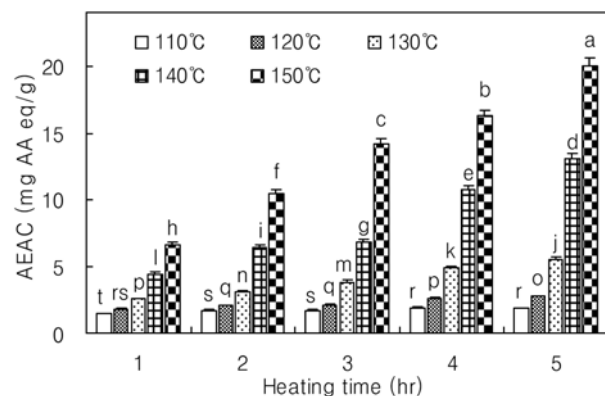


**Fig. 4.** DPPH radical scavenging activity of 20% sucrose solution treated with high temperature and high pressure. (untreated sucrose solution:  $0.00 \pm 0.00\%$ ).

by furfural and 5-methylfurfural. Antal *et al.* (38) reported that HMF was formed during the sucrose decomposition and the low pH promoted the formation of HMF and successive fragmentation to levulinic acid, formic acid and so on.

**DPPH radical scavenging activity** DPPH radical scavenging activities (electron donating ability; EDA) of 20% sucrose solution with heating are shown in Fig. 4. Several researchers have indicated that heat treatment causes enhanced antioxidant activity in fruits and vegetables, because of the enhancement of the antioxidant properties of naturally occurring compounds or the formation of novel compounds, such as Maillard reaction products, that have antioxidant activity (39,40). Yen and Hsieh (41) reported that xylose and lysine Maillard reaction products showed dose-dependent scavenging activity for the hydroxyl radical, which might have been attributed to the combined effects of reducing power, donation of hydrogen atoms, and scavenging of reactive oxygen species. Significant differences in the EDA of heated sucrose solution were observed with heating ( $p < 0.001$ ; Table 3). The EDA of heated sucrose solution increased significantly with increasing heating temperature (110 to 150°C) and time (1 to 5 hr;  $p < 0.001$ ). The EDA of the unheated sucrose solution and heated sucrose solution below 110°C for 4 hr at a concentration of 1 g/mL is not shown. The EDA slowly increased with heating up to 140°C for 2 hr, and then suddenly increased after that. The EDA was 12.75 and 21.11% at 140°C for 2 and 3 hr, respectively. The EDA increased at 150°C for 5 hr, and the maximum EDA was 93.23% at 150°C for 5 hr. The EDA after heating to 140°C suddenly increased, because fructose and glucose were rapidly generated by the thermal degradation of sucrose (Fig. 2). Thermally, sucrose is first degraded to fructose and glucose, which then break down to heat degradation products. Such heat degradation products have been reported to have antioxidant activity, through scavenging oxygen radicals or chelating metals (42).

**ABTS cation radical scavenging activity** ABTS cation radical scavenging activity (AEAC) of the 20% sucrose solution with heating is shown in Fig. 5. Yilmaz and



**Fig. 5.** ABTS cation radical scavenging activity (AEAC) of 20% sucrose solution treated with high temperature and high pressure. (untreated sucrose solution:  $1.50 \pm 0.02$  mg AA eq/g).

Toledo (42) reported that Maillard reaction products (MRPs) had antioxidant activity. MRPs, especially melanoidins, have been reported to have antioxidant activity through scavenging oxygen radicals or chelating metals. Osada and Sibamoto (43) also reported that MRPs inhibited oxidation in model systems, as well as in storage experiments with food products. Significant differences in the AEAC of heated sucrose solution were observed with various heating conditions ( $p < 0.001$ ; Table 3). The AEAC of heated sucrose solution increased significantly with increasing heating temperature (110 to 150°C) and time (1 to 5 hr;  $p < 0.001$ ). The AEAC slowly increased with heating to 140°C, and then suddenly increased after that. The AEAC increased to 150°C for 5 hr, and the maximum AEAC was 20.03 mg AA eq/g at 150°C for 5 hr.

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