

# Replacement of Chlorine Treatment for Cake Flour<sup>1</sup>

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## ABSTRACT

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Treatment of unchlorinated soft wheat flour at 7% flour moisture for 30 min at 125°C, and supplementing that flour with 0.12% (fwb) xanthan gum, produced cakes with volumes that were slightly greater than those of cakes made with the chlorinated control flour. The crumb grains were essentially equivalent. The addition of 200 or 300 ppm L-cysteine to cake batter made with unchlorinated, heat-treated flour also produced cakes with volumes and crumb grain scores equal to those of cakes made with the chlorinated control flour. Addition of hydrogen

peroxide plus peroxidase to the cake formula containing heat-treated flour also improved the cake volume, grain, and color. However, the volume was less than that of the cakes made with the chlorine-treated control flour. Heat treatment also reduced the degree of shrink of the cake during baking to a value equal to that of the cakes made with the chlorinated control flour. Enthalpy values of heat-treated flour measured by differential scanning calorimetry showed that starch was not gelatinized during heating.

Chlorination of soft wheat flour was introduced in the early 1930s and is necessary to produce high-ratio cakes with optimum quality characteristics (Pylar 1988). Although the exact mechanisms are still uncertain, cakes baked from optimally chlorinated flour at optimum absorption levels have improved volume, a finer more uniform grain, whiter crumb color (Sollars 1958), improved symmetry (Gough et al 1978), and improved sensory properties (Gaines and Donelson 1982). The chlorine gas reacts with the flour in a rapid, surface-dependent reaction (Yamazaki and Kissell 1978). Essentially all flour components (gluten, starch, lipids, water solubles, and pentosans) are modified chemically during the reaction (Stauffer 1990). The use of chlorine gas in a flour mill poses safety concerns. In addition, the public is concerned about the use of certain chemicals in food processing. Both of these point to the need to find a safe replacement for chlorine to treat cake flours.

Russo and Doe (1970) reported that heat treatment of unchlorinated cake flour improved its cake-baking properties. They found the optimum treatment temperature to be 120°C, but concluded that the time of treatment was not important. Flour baking performance was improved when the moisture level of the flour was reduced <7% during treatment. Although the flour's cake-baking quality improved compared to that of the untreated control, the quality of the cakes was not equal to the quality of cakes made with the chlorinated control. The authors noted that unspecified formulation changes were necessary to produce a "satisfactory" cake. A recent patent (Rabe and Meyers 1995) details certain formulation changes that are beneficial.

Johnson and Hosene (1980) studied the effect of heating and storage on the functional qualities of flour. Flour stored at room temperature for two months resulted in cakes with improved volume and grain. These age-related changes were accelerated by heat. Heat treatment of unaged flours improved the crumb grain and eliminated collapse during baking.

The purpose of this study was to develop a method of heat treatment that produced flour with baking qualities equal to or better than those produced by chlorination.

## MATERIALS AND METHODS

### Materials

The flour used in this study was a commercially milled unchlorinated cake flour obtained from General Mills (13.3% moisture, 0.38% ash, 7.93% protein, 5.65 pH). A subsample of the flour was chlorinated using the procedure and apparatus described by Kissell and Marshall (1972). Samples were chlorinated to several pH values. The subsample chlorinated to pH 5.1 produced the best cake volume and grain and was selected as the optimum treatment.

Sugar was obtained from Amstar Sugar Corp., New York, NY. Cake and icing shortening, a partially hydrogenated shortening with mono- and diglycerides, was obtained from Kraft Food Ingredients Corp. Jacksonville, IL. Dried egg whites were from Monark Egg Corp., Kansas City, KS. Salt was from Fisher Scientific Co., St. Louis, MO. High-heat nonfat dried milk was acquired from ADM Arkady, Olathe, KS.

Levin lite (sodium aluminum phosphate [SALP] 27.5%), monocalcium phosphate (MCP, 12.5%), sodium bicarbonate (37.5%), and redried starch (22.5%) were blended to make baking powder. The SALP and MCP were obtained from Monsanto (St. Louis, MO). Redried starch was made by drying corn starch (American-Maize Products, Hammond, ID) in a convection oven at 100°C to 2% moisture.

Food-grade xanthan gum (Keltrol TF) was obtained from Kelco, Chicago, IL. L-cysteine and peroxidase (horseradish) were from Sigma Chemical Co., St. Louis, MO. Hydrogen peroxide (30%) was obtained from Aldrich Chemical, Milwaukee, WI.

### Baking Method

White layer cakes were baked according to method 10-90 (AACC 1983). Absorption was optimized for chlorinated and unchlorinated controls as well as for each heat-treated sample. Optimum absorption was taken as that level which produced the best volume and crumb grain. Cakes were baked at 191°C for 25 min in a reel oven from National Mfg. (Lincoln, NE). Cake volumes were calculated based on measurements from a template according to method 10-91 (AACC 1983). A subjective scale based on the following parameters was devised to score the crumb grain: very fine, uniform grain = 10; uniform with a few slightly larger uniform cells = 9; large cells and nonuniform grain = 5; and an extremely poor grain and tunneling = 1.

### Adjustment of Flour Moisture

Two methods were used to lower the moisture content of the unchlorinated flour before heat treatment. For flour moisture

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<8%, the flour was placed in a cotton cloth and freeze-dried to the required moisture. For flour moisture >8%, the flour was spread evenly in an aluminum pan and dried in a convection oven at ≈35°C until the desired moisture was obtained.

### Heat Treatment

The flour was spread evenly (≈1.0 cm thick) in a 43- × 30.5- × 4-cm aluminum pan and heated in a convection oven. After heat treatment, the samples were rehydrated to ≈13% moisture in a fermentation cabinet set at 85% rh before baking.

### Moisture Content

Moisture content was determined according to method 44-15A (AACC 1983). Samples were allowed to equilibrate at least overnight before moisture was determined.

### Xanthan Gum

Xanthan gum was added with the dry ingredients at levels of 0.48, 0.24, and 0.12% (fwb) to optimize usage level. Water absorption was optimized at each level. The flour used was heat-treated at 7% moisture for 30 min at 125°C.

### Cysteine

L-Cysteine was added with the dry ingredients at levels of 100, 200, and 300 ppm (fwb). The flour used was heat-treated at 7% moisture for 30 min at 125°C.

### Hydrogen Peroxide Treatment

Hydrogen peroxide (30%) was added to the cake batter at levels of 0.05, 0.10, and 0.20% (fwb). The flour used was heat-treated at 7% moisture for 30 min at 125°C. Peroxidase (horseradish) was added at 380 units/200 g of flour to replace the flour's native peroxidase, which was assumed to be inactivated during heat treatment.

### Differential Scanning Calorimetry

A Perkin-Elmer DSC-2 with an Intracooler-II system was used to determine the enthalpy of starch gelatinization. This was to determine whether any starch gelatinization had occurred during heat treatment. An empty pan was used for the reference. Samples were heated in the calorimeter at 10°C/min with a sensitivity of 0.5 mcal/sec.

### Height Increase During Baking

An aluminum ruler was held suspended by a detachable frame that clipped to the sides of a cake pan. The ruler was centered 8 mm above the base of the pan, to prevent heat transfer from the

pan bottom to the ruler and minimize premature setting of the cake around the ruler. The increase in cake height during baking was monitored by taking photographs at 1-min intervals with a camera mounted to the oven door. Batter temperature during baking was determined by a thermocouple suspended in the batter. The cake height during baking then was plotted as a function of batter temperature. Shrink during baking was measured as the difference in cake height at its maximum and final heights.

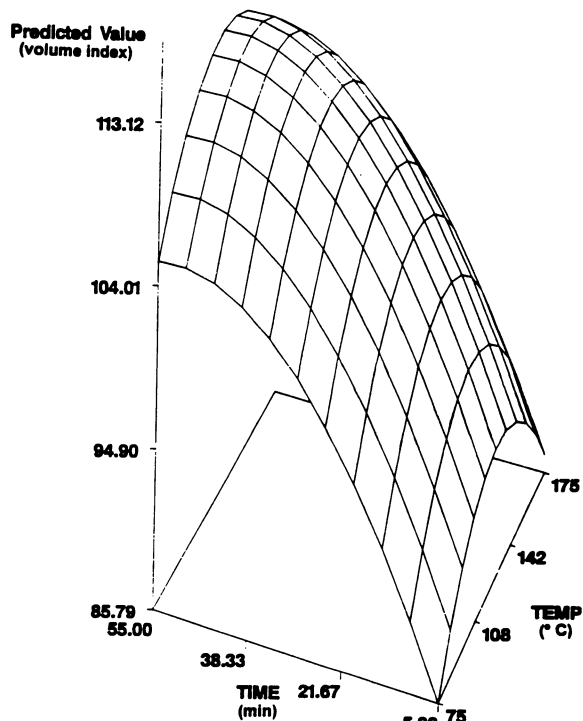


Fig. 1. Response surface graph showing the predicted cake volume index as a function of treatment temperature and time at 1% flour moisture.

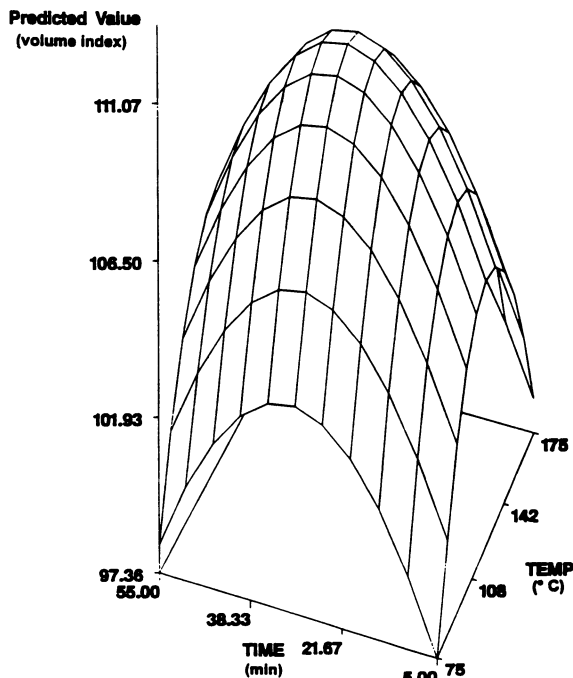


Fig. 2. Response surface graph showing the predicted cake volume index as a function of treatment temperature and time at 4% flour moisture.

TABLE I  
Moisture Content, Time, and Temperature Combinations  
Tested in RSM Experimental Design

Moisture (%)	Time (min)	Temperature (°C)
2	15	100
2	45	100
2	15	150
2	45	150
6	15	150
6	45	100
6	15	100
6	45	150
4	5	125
4	30	125 <sup>a</sup>
4	55	125
4	30	83
4	30	167
0.64	30	125
7.4	30	125

<sup>a</sup> This treatment (the center point) was replicated six times. All other treatments were replicated once.

### Batter Viscosity During Baking

Batter viscosity was determined using an oscillating rod viscometer (model 710, Nametre Co., Metuchen, NJ) in combination with the electrical resistance oven-baking technique described by Shelke et al (1990).

### Experimental Design and Statistical Analysis

Response surface methodology (RSM) was employed to determine the optimum combination of moisture, time, and temperature for heat treatment of flour. The experimental design described by Cockran and Cox (1957) for three variables with five levels of each was used (Table I). The center point (4%, 30 min, 125°C) was replicated six times. All other treatments were

replicated once. All heat-treatments were performed and baked in random order. Data were analyzed using the response surface regression procedure (SAS Institute, Cary, NC). Surface response plots were generated from the best model as determined by SAS. Additional data were evaluated by analysis of variance and least significant difference using SAS.

## RESULTS AND DISCUSSION

Preliminary work examining the effect of heat treatment on unchlorinated soft wheat flour was used to determine parameters and limits for the response surface study. Baking results indicated that heat treatment at normal moisture levels (13%) improved cake volume, but lowered cake crumb quality (score). Heat treatment at these moisture levels may result in protein modification or possibly starch gelatinization. Therefore, the moisture content of the flour was reduced before heating in an effort to prevent these changes from occurring and to allow heat treatment to have a beneficial effect on both volume and crumb grain. Heating at both 100 and 150°C combined with lower flour moisture levels (2–3%) resulted in improved cake volumes and crumb scores. Heat treatment at 200°C charred the flour and rendered it unsuitable for baking. Although heat treatment at reduced moisture levels improved both cake volume and crumb score compared to those of the untreated control, the values were not equivalent to those of the cakes made with chlorinated flour.

### Response Surface Study

An RSM design was employed to determine the optimum combination of the three parameters (flour moisture, treatment time, and treatment temperature) thought to affect heat treatment. Computer-generated response surface graphs for 1, 4, and 7% moisture levels are shown in Figures 1–3, respectively. Comparison of the graphs' maxima show that predicted volumes increased as flour moisture level increased. Volumes of cakes made with all heat-treated samples were greater than the volume of the cake made with the untreated control, but less than that of the cake made with the chlorine-treated control. Thus, moisture content of the flour is an important parameter affecting heat treatment. However, optimum treatment temperature ( $\approx 125^\circ\text{C}$ ) remained constant for all moisture levels.

Time was also a major factor affecting heat treatment. This was evident by comparing the time at which each surface reached optimum volume. At 1% flour moisture, optimum volume was not achieved until treatment time exceeded 55 min. As moisture

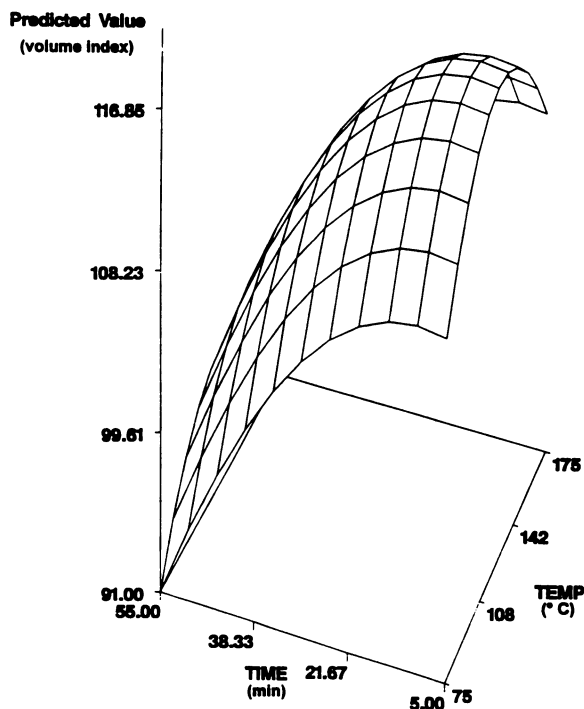


Fig. 3. Response surface graph showing the predicted cake volume index as a function of treatment temperature and time at 7% flour moisture.

TABLE II  
Effect of Heat Treatment at 150°C with 8, 9, and 10% Flour Moisture on Water Absorption, Volume Index, and Grain Score

Heat Treatment		Absorption (%)	Volume Index <sup>a</sup>	Grain Score <sup>b</sup>
Moisture (%)	Time (min)			
8	5	120	110 ef	5
8	5	130	114 cde	8 <sup>c</sup>
8	10	120	113 cde	5
8	10	130	118 ab	9 <sup>c</sup>
9	5	130	108 f	8
9	5	140	111 def	10 <sup>c</sup>
9	10	130	115 bcd	9
9	10	140	113 cde	10 <sup>c</sup>
10	5	120	111 def	5
10	5	130	116 bc	9 <sup>c</sup>
10	10	120	115 bcd	5
10	10	130	120 a	9 <sup>c</sup>
Chlorinated control		130	120 a	9
Untreated control		120	94 g	5

<sup>a</sup> Means in a column with the same letter are not significantly different ( $P = 0.05$ ).

<sup>b</sup> Score of 1 indicates extremely poor grain; score of 10 indicates excellent grain.

<sup>c</sup> Cakes unsatisfactory because surface was pitted.

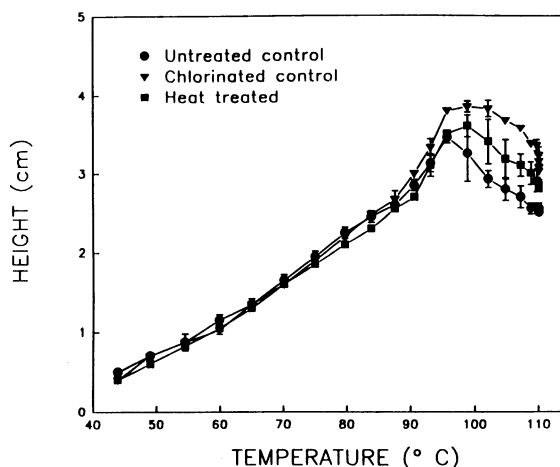


Fig. 4. Effect of heat treatment on cake height increase during baking; chlorinated control = 5.1 pH, 130% water absorption; untreated control = 5.65 pH, 120% absorption; and heat treated = 7% flour moisture, 125°C, 30 min, 140% absorption. Error bars indicate standard deviation.

content increased, the time required to obtain optimum volume decreased to 10 min at 7% flour moisture. Thus, a significant interaction occurred between time and flour moisture. The presumed action of heat treatment of flour was to accelerate the oxidation reactions that occur naturally during storage or chlorine treatment. These results suggest that higher moisture content during heating increased the rate or the extent of the reactions, thereby shortening treatment time.

### Heat Treatment at Increased Flour Moisture

Heat treatment at increased flour moisture levels was investigated to determine at what point flour moisture content became detrimental to cake quality. Heat treatment at 8, 9, and 10% moisture resulted in improved cake volume (Table II). In some cases, the volume index and crumb score were comparable to those of cakes made with the chlorinated control; however, the appearance of the cakes was unsatisfactory because the surfaces were pitted. We assumed this was caused by excess water absorption. Lowering water absorption minimized the pitting but decreased both volume and grain scores.

### Differential Scanning Calorimetry

A calorimeter was used to determine the effect of flour moisture and temperature on starch during heat treatment. Enthalpy values did not vary significantly between heat treatments (data not shown). Thus, starch gelatinization did not occur during heat treatment.

### Height Increase During Baking

Height increases of cakes baked with chlorinated control, untreated control, and heat-treated flours were monitored to determine whether differences occurred during baking. During the early and intermediate stages of baking, rates of increase in cake height were similar for all three samples (Fig. 4). At 95°C, cakes baked with the chlorinated control flour had a significantly higher maximum expansion, whereas the cake made with untreated control flour collapsed. Cakes baked with heat-treated flour reached a maximum height between those of the cakes made with chlorine-treated flour and those made with the untreated control. Cakes made with heat-treated and chlorine-treated flours had similar degrees of shrinkage during baking (0.80 and 0.82 cm, respectively). Both of these were significantly less than shrinkage of cakes made with the untreated control flour (1.00 cm).

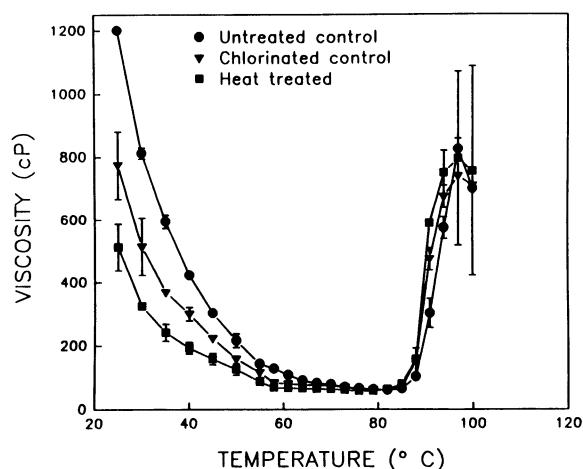


Fig. 5. Effect of heat treatment on cake batter viscosity during electrical resistance oven baking; chlorinated control = 5.1 pH, 130% water absorption; untreated control = 5.65 pH, 120% absorption; and heat treated = 7% flour moisture, 125°C, 30 min, 140% absorption. Error bars indicate standard deviation.

### Batter Viscosity During Baking

The viscosity profile (Fig. 5) of cake batters during baking shows that the viscosity was much lower at room temperature with heat-treated flour than with the untreated and chlorine-treated controls. Viscosity of batter made with heat-treated flour reached a lower minimum at a lower temperature than that of batter made with either the untreated or chlorinated controls during baking. Lower batter viscosity allows large gas cells to migrate to the surface and be lost, resulting in a fine, uniform crumb grain. This crumb grain is reflected in the high grain score of the cakes baked with heat-treated flour. The higher viscosity of the batter made with untreated flour would reduce the migration of the gas cells. Coalescence of gas cells results in larger cells that are retained by the viscous batter, producing cakes with an open crumb grain.

Both the heat-treated and chlorinated control flours produced batters that showed a rapid increase in viscosity between 85–94°C (Fig. 5). Batters made with the untreated control had a significantly lower viscosity at both 91 and 94°C. This difference in viscosity may be related to the cake's ability to withstand the transformation from a foam to a sponge and to the degree of shrink during baking. Batter made with the untreated flour increased in viscosity more slowly and collapsed significantly more than batter made with either the heat-treated or chlorinated control flour.

### Addition of Xanthan Gum

Viscosity profiles showed that the viscosity of the batter from heat-treated flour was much lower at room temperature than that

TABLE III  
Effect of Xanthan Gum on Water Absorption, Volume Index, and Grain Score of Cakes Made With Chlorine-Treated, Untreated, and Heat-Treated Flours

Sample	Xanthan Gum (%)	Absorption (%)	Volume (%) <sup>a</sup>	Grain Score <sup>b</sup>
Chlorinated control	0.00	130	121 c	9
Untreated control	0.00	120	98 f	5
Heat-treated	0.00	140	113 d	10
Chlorinated control	0.24	130	125 a	7
Untreated control	0.24	120	115 d	5
Heat-treated	0.24	130	126 a	4
Chlorinated control	0.12	130	120 c	8
Untreated control	0.12	120	115 d	5
Heat-treated	0.12	130	125 a	8
Chlorinated control	0.06	130	124 ab	9
Untreated control	0.06	120	110 e	5
Heat-treated	0.06	130	121 bc	9

<sup>a</sup> Means in a column with the same letter are not significantly different ( $P = 0.05$ ).

<sup>b</sup> Score of 1 indicates extremely poor grain; score of 10 indicates excellent grain.

TABLE IV  
Effect of Cysteine on Volume Index and Grain Score of Cakes Made With Heat-Treated Flour

Sample	Cysteine (ppm)	Absorption (%)	Volume Index <sup>a</sup>	Grain Score <sup>b</sup>
Chlorinated control	0	130	120 a	9
Untreated control	0	120	95 c	5
Heat-treated	0	130	113 b	10
Heat-treated	100	130	113 b	9
Heat-treated	200	130	121 a	9
Heat-treated	300	130	121 a	9

<sup>a</sup> Means in a column with the same letter are not significantly different ( $P = 0.05$ ).

<sup>b</sup> Score of 1 indicates extremely poor grain; score of 10 indicates excellent grain.

**TABLE V**  
Effect of Hydrogen Peroxide Plus Peroxidase  
on the Properties of Heat-Treated Flour

Sample	Hydrogen Peroxide (%)	Peroxidase Units <sup>a</sup>	Volume Index <sup>b</sup>	Grain Score <sup>c</sup>
Chlorinated control	0.00	0	120 a	9
Untreated control	0.00	0	110 bc	5
Heat-treated	0.00	0	108 c	10
Heat-treated	0.05	380	114 ab	9
Heat-treated	0.10	380	114 ab	9
Heat-treated	0.20	380	113 bc	9

<sup>a</sup> Per cake batter made from 200 g of flour.

<sup>b</sup> Means in a column with the same letter are not significantly different ( $P = 0.05$ ).

<sup>c</sup> Score of 1 indicates extremely poor grain; score of 10 indicates excellent grain.

of batter from the untreated or chlorine-treated controls (Fig. 5). Therefore, xanthan gum was added to increase the batter viscosity. The addition of xanthan gum increased not only batter viscosity but also cake volume (Miller and Hosenev 1993). Addition of xanthan gum to batters made with heat-treated flours also gave increased cake volume (Table III). The volume increase was much greater for cakes made with the heat-treated flour than for cakes made with either the chlorine-treated or untreated controls. The cake volume with heat-treated flour plus xanthan (0.12%) was equal to or greater than the cake volume with the chlorine-treated flour either with or without xanthan. Lower levels of xanthan slightly decreased crumb scores for cakes made with both the heat-treated and chlorine-treated samples, whereas higher levels of xanthan resulted in poor crumb grain with large holes and tunnels. Thus, the combination of heat treatment and xanthan could replace chlorine treatment.

#### Addition of L-Cysteine

One possible reason for the lower batter viscosity at room temperature is that the proteins were polymerized during heat treatment and became less soluble. L-Cysteine was added to determine whether depolymerization had an effect on the resultant cake. The addition of cysteine to cakes made with heat-treated flour increased volume and improved crumb grain scores to levels equal to those of cakes made with the chlorinated control (Table IV). Apparently, polymerization of the proteins resulted in a cake structure that was too rigid and prevented the cake from expanding to a maximum volume. Thus, heat treatment plus cysteine could replace chlorine treatment for cake flours.

#### Treatment with Hydrogen Peroxide

Hydrogen peroxide is known to increase the viscosity of flour water suspensions (Durham 1925). We assumed that this increase in viscosity might allow hydrogen peroxide to replace the more expensive xanthan gum. The increase in viscosity (oxidative gelation) of the water-soluble fraction of flour is known to involve the enzyme peroxidase (Hosenev and Faubion 1981). The heat treatment of the flour was assumed to have denatured the indigeneous peroxidase. Therefore, both hydrogen peroxide and peroxidase were added to the heat-treated flour (Table V). This treatment reduced the surface pitting in the crust and greatly improved the grain. Cakes containing hydrogen peroxide also had a desirable white crumb color. Although the volume index increased from 107 to 114, it was lower than that of cakes made with the chlorine-treated flour. Neither higher levels of hydrogen peroxide nor holding the batter for 20 min before baking to allow greater enzyme activity produced larger cakes (data not shown).

## CONCLUSIONS

Heat treatment of flour improved cake volume compared to the untreated control. At certain moisture levels before treatment (8–10%), the cake volume index approached that of the chlorine-treated control. However, these cakes were unsatisfactory because of pronounced surface pitting. In contrast, the crumb grain scores were higher than for either the untreated or chlorinated controls.

The viscosity of batter made from the heat-treated flour was much lower than for either the untreated or chlorinated control batters. Addition of xanthan to the batter made with heat-treated flour gave cakes that were equal to or better than those from chlorine-treated flour. Because xanthan is a relatively expensive ingredient, hydrogen peroxide and peroxidase were used to increase the viscosity of the batter. The volume, grain, and color were all improved when compared to the untreated flour. However, the volume was not equal to the chlorine-treated control.

Proteins are known to polymerize during heating and become less soluble. Therefore, L-cysteine was added to batters made with heat-treated flour. This produced cakes with volumes and crumb grain scores equal to those of the chlorinated control.

Thus, chlorine treatment of cake flour can be replaced by heat treating the flour (7% moisture, 30 min, 125°C) and adding either xanthan gum (0.12%) or L-cysteine (200 ppm) to the formula. Hydrogen peroxide plus peroxidase added to the batter made with heat-treated flour gave crumb grain and color equal to those of the chlorine-treated flour and only slightly lower volume indexes.

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