

# Correlation Between the Glass-Transition Point and Color Change of Heat-Treated Gluten

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ABSTRACT

Cereal Chem. 66(4):268-270

The color change and the density of heat-treated wheat gluten varies, depending on the treatment conditions. We studied heat treatment of gluten by comparing color changes in samples treated under various conditions. The change in color was dependent on the treatment temperature, up to a critical temperature specific to the sample. The critical temperature was significantly increased when the moisture content of the sample was reduced. For example, the critical temperature at 13% moisture was 30°C, whereas the critical temperature at 5% moisture was 100°C. Below the critical temperature, the degree of color change was found to be

proportional to the treatment temperature. The density of heat-treated wheat gluten was proportional to the logarithm of the change in color. Based on differential scanning calorimetric measurement of wheat gluten at various moisture levels, it was confirmed that the critical temperature determined by the color change corresponded to the glass transition temperature of the sample. In fact, wheat gluten was transformed into a glassy material by the heat treatment, indicating the creation of noncrystalline (amorphous) molecular orientations.

Wheat gluten is a biological polymer. A polymer can be softened to form a semiliquid, or rubbery substance, by elevating its temperature, and it will change into a glassy solid or a plastic (bioplastic) by subsequent cooling. The molecular motion of an amorphous polymer under low temperature is believed to be frozen in random configuration, thus rendering it glassy and immobile. When thermal energy is applied to such a polymer, the molecular motion is reactivated and the molecules start sliding past one another. At this point, the polymer starts to exhibit a viscous, rubbery, or flexible nature. This type of physical change has been called a "glass transition" (Hoseney et al 1986).

It has been reported by some researchers (Arntfield 1981, Eliasson and Hegg 1980) that wheat gluten shows an extremely small or no denaturing peak when it is heated in a differential scanning calorimeter within the 30–130°C range. Hoseney et al (1986) examined a commercial wheat gluten by differential scanning calorimetry (DSC) and reported that the glass-transition point varied as a function of water content. They concluded that wheat gluten consists of amorphous random polymers, and that it contains virtually no ordered long-chain molecules.

In this study, a wheat gluten (amorphous biopolymer) was heat treated at various temperatures and moisture contents. Changes in the color of the heat-treated wheat gluten were studied and correlated with the glass-transition point as measured by DSC.

## MATERIALS AND METHODS

### Raw Materials and Sample Preparation

Wheat gluten was purchased from Ishizu Pharmaceutical Co. It contained 4.5% moisture, 70.7% crude protein determined by the Kjeldahl method ( $N \times 5.7$ ), and 2.8% extractable lipids.

Moisture content of the wheat gluten was adjusted to a desired level by adding a calculated amount of ice powder to the sample and grinding at  $-20^\circ\text{C}$  as described previously (Fujio et al 1988). The final moisture contents of the samples were 5, 8, 11, and 13%.

### Heat Treatment Under Pressure

A Koka flow tester (Shimadzu Seisakusho Co., model 61323) was used for the heat treatment of wheat gluten under pressure. The profile of the flow tester and the treatment procedures have been described previously (Fujio et al 1988). By a similar procedure, the samples were heat treated at various temperatures between 10 and  $120^\circ\text{C}$  under a pressure of  $200\text{ kg/cm}^2$ .

### Measurement of Color Change

The color changes in heat-treated gluten samples were measured with a Minolta Chroma meter CR-100 (Minolta Camera Co.) using true daylight. The Chroma meter was calibrated with a standard white tile. The unit of change in sample color was expressed by  $\Delta E$  (color difference using the  $L$ ,  $a$ ,  $b$  colorimetry system). An untreated gluten sample was used as the reference for determining the  $\Delta E$  change in color. A sample piece was placed on the standard white tile, and its surface color was measured by the Chroma meter equipped with a silicon photocell. The color change was determined by reference to data stored in the memory of the instrument.

### Density Measurement

Samples removed from the flow tester, after being heated and subsequently cooled, were rod shaped, 11.3 mm in diameter ( $1.0\text{ cm}^2$  cross-sectional area). Therefore, the densities of the treated samples were determined by simply measuring the lengths and the weights of the rod-shaped samples.

### DSC

Samples of the wheat gluten were heated in a differential scanning calorimeter (Seiko DSC-100, Seiko Electric Co.) to determine the incremental changes in heat capacity, " $C_p$ ," which is an indication of the glass-transition point. The instrument was

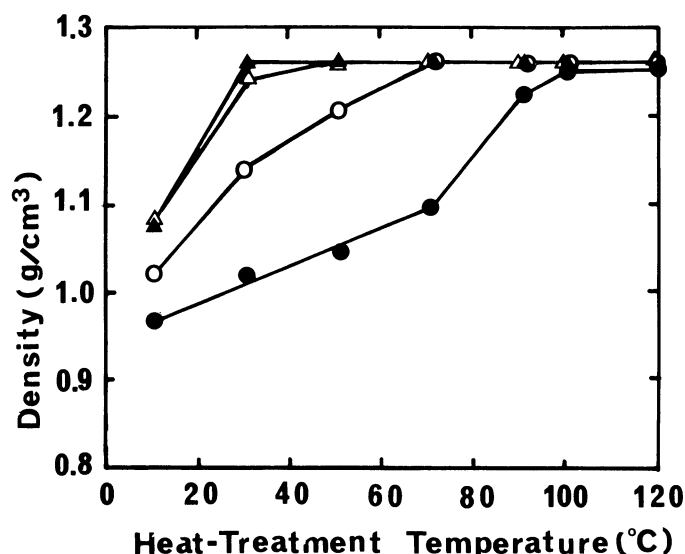


Fig. 1. Effects of heat treatment temperature on density of wheat gluten at  $200\text{ kg/cm}^2$  pressure and various moisture contents: 5%  $\text{H}_2\text{O}$  (●), 8%  $\text{H}_2\text{O}$  (○), 11%  $\text{H}_2\text{O}$  (△), 13%  $\text{H}_2\text{O}$  (▲).

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calibrated with elemental indium. Sample temperatures were increased at 10°C/min from 10 to 170°C. An empty pan was used as a reference. The glass-transition point was determined by finding the midpoint of the transition in heat capacity, Cp.

## RESULTS AND DISCUSSION

### Density and Color Change

Figure 1 shows the change in density of wheat gluten treated at various temperatures and moisture contents under 200 kg/cm<sup>2</sup> pressure. At a moisture content of 5%, the density of the sample treated at 10°C (corresponding to the native gluten) was about 0.95 g/cm<sup>3</sup>. The samples became more compressible as the moisture content was increased, resulting in a higher density. The density also increased significantly when the treatment temperature was increased, and it reached a constant at about 1.26 g/cm<sup>3</sup>. The critical temperature, defined as the temperature at which the density reached a constant value, decreased with increasing moisture content during treatment. These observations show that, the higher the temperature and moisture content during heat treatment, the greater the compressibility of wheat gluten. This results in a constant density after the cooling step.

As shown in Figure 2, the color also changed with temperature and moisture content, similar to the change found for density. For example, the color change reached a constant value at the critical temperature. The critical temperature decreased with increasing moisture content. The critical temperature obtained by the measurement of color change coincided with the critical temperature obtained by density measurement.

It was observed that a gluten sample cooled after heat treatment at a temperature above the critical temperature had a glassy texture and a dark brown color. It was also observed that a thin slice of such a sample was translucent. When the moisture content was above 13%, this phenomenon occurred even at room temperature. Therefore, it can be concluded that the change in color of the heat-treated gluten occurred because of the change in density resulting from compression under the pressure. The relationship between the change in density and the change in color is shown in Figure 3. The density of the heat-treated gluten samples showed good correlation with the logarithm of the change in color.

### Correlation Between Glass-Transition Point and Change in Color

Figure 4 shows the changes in the glass-transition temperature (T<sub>g</sub>, change in heat capacity) measured by DSC as a function of moisture content. The wheat gluten in this study showed only a small change in heat capacity and showed no denaturation peak when the moisture content was below 16%. According to polymer

theory (Ferry 1980, Jin et al 1984), the water contained in a polymer-diluent system acts as a plasticizer and depresses the glass-transition temperature. As expected, the glass-transition temperature varied with the moisture content of the gluten samples. The glass-transition temperature, T<sub>g</sub>, decreased with increasing moisture content, which concurred with the results reported by Hosney et al (1986).

In Figure 4, the critical temperatures obtained by the density measurements and the critical temperatures obtained by color change measurements were plotted against moisture content. The good agreement indicates that the critical temperatures shown in Figures 2 and 3 are actually the glass-transition temperatures at

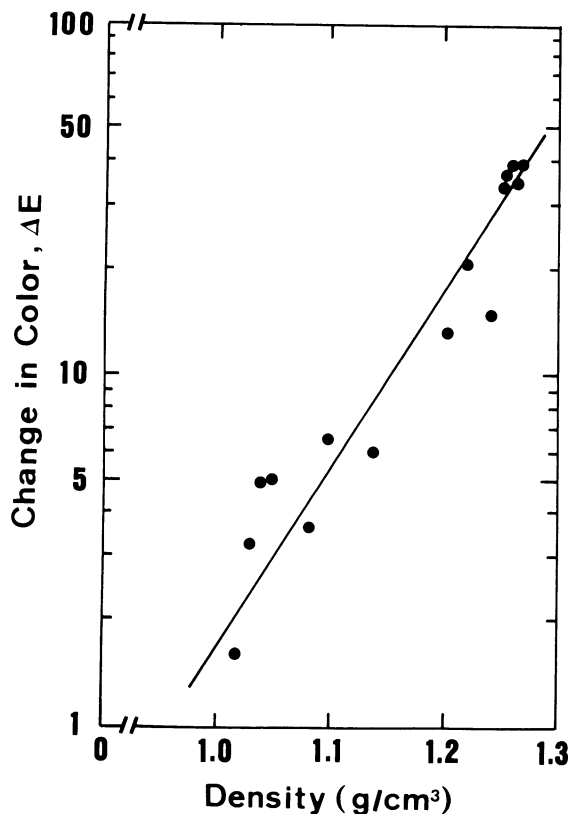


Fig. 3. Correlation between color change and density of wheat gluten under pressure at 100 kg/cm<sup>2</sup>.

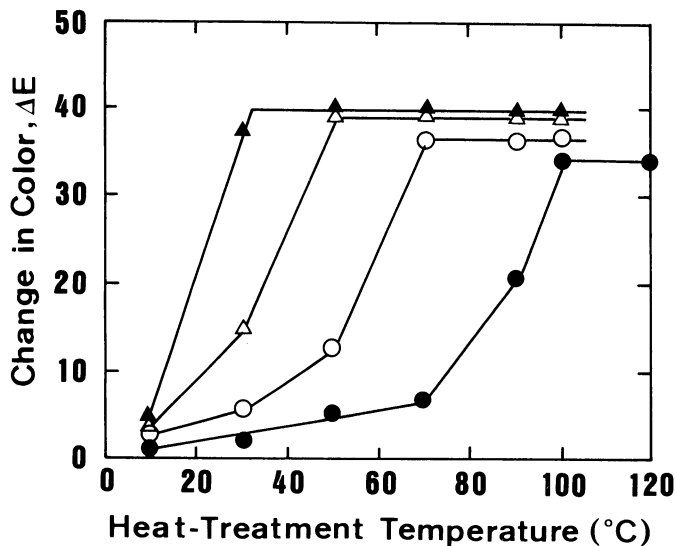


Fig. 2. Effect of heat treatment temperature on the change in color under pressure at 200 kg/cm<sup>2</sup>: 5% H<sub>2</sub>O (●), 8% H<sub>2</sub>O (○), 11% H<sub>2</sub>O (Δ), 13% H<sub>2</sub>O (▲).

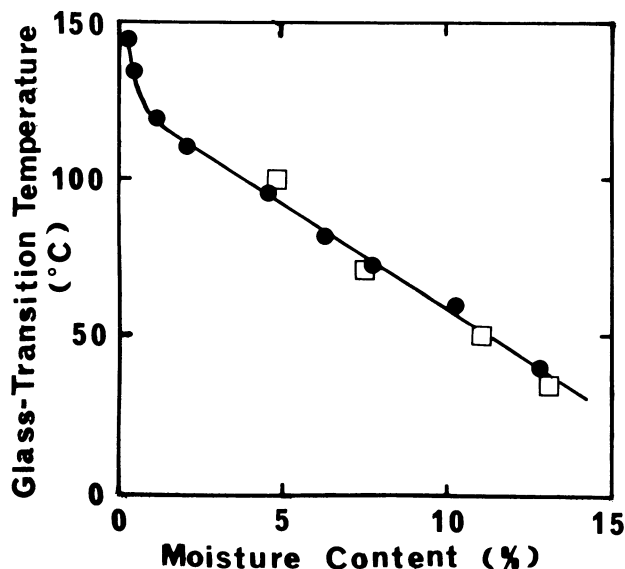


Fig. 4. Effects of moisture content on the glass-transition temperature: glass-transition temperature measured by differential scanning calorimetry (●), critical temperature from color change (□).

various moisture contents. It is thought that the wheat gluten is transformed into a rubbery, flexible material when it is heated above its glass-transition temperature under a constant pressure, and then into a high-density, glassy material when it is subsequently cooled. It has been reported that the refractivity changes, according to the changes in its specific volume (Nielsen 1962). This change in refractivity, in turn, changes the color of the wheat gluten when it is heated above its glass-transition point.

### CONCLUSIONS

Based upon the observations made in this study, the following conclusions can be drawn. The glass-transition phenomenon contributes to a color change in heat-treated wheat gluten through a change in its refractivity. Therefore, the glass transition of a wheat gluten can be detected by monitoring the change in color when it is being heated under pressure. Wheat gluten may be considered to be an amorphous biopolymer, which explains the occurrence of the glass transition and the absence of a denaturation peak when it is examined by DSC.

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[Received July 7, 1988. Revision received January 14, 1989. Accepted January 16, 1989.]