# Glass Transition Temperature of Rice Kernels Determined by Dynamic Mechanical Thermal Analysis

T. J. Siebenmorgen, W. Yang, Z. Sun

**Abstract.** *A protocol was developed to measure the glass transition temperature ($T_g$) of brown rice kernels using dynamic mechanical thermal analysis (DMTA). In order to accommodate the sample requirements of the dynamic mechanical thermal analyzer and prevent moisture loss during experiments, kernels were first wrapped with a thin layer of parafilm and then a thin layer of aluminum foil. The wrapped kernels were then filed at both ends with fine sandpaper to produce 3 mm long rice cylinders with parallel ends. The dynamic mechanical properties of brown rice kernels were determined over a temperature range of −40°C to 120°C. During each DMTA test, the onset temperature of glass transition was measured as the $T_g$ of a brown rice kernel, since moisture diffusion and other physical properties begin to change drastically above the onset temperature. No dramatic changes in both the storage modulus (E′) and the loss modulus (E″) of brown rice kernels were found around $T_g$, indicating that relaxation of the amorphous phase of the rice kernel was quite limited. However, E″/E′ (tanδ) showed a much better transition for brown rice kernels, and thus the E″/E′ vs. temperature curves were used to determine $T_g$ values of rice kernels. It was found that the $T_g$ values of rice kernels decreased linearly with increases in moisture content (MC) within the tested range. The $T_g$ of rice kernels was not affected by variety.*

***Keywords.*** *Glass transition, Rice properties, Thermomechanical properties.*

Glass transition temperature is perhaps the most important single parameter that determines the application of many polymers. Many physical properties, including specific heat, specific volume, expansion coefficients, and viscoelasticity, change as polymers transition from a "glassy" to "rubbery" state (Ferry, 1980; Rabek, 1980). Application of the glass transition concept in food science has attracted much attention over the last 20 years, since food components, such as starch and proteins, are biopolymers. State diagrams have been applied to predict changes in food properties below and above the glass transition temperature ($T_g$) (Slade and Levine, 1991; Zeleznak and Hoseney, 1987).

Rice is usually harvested around 16% to 22% w.b. (wet basis) moisture content (MC) in the U.S. and dried to approximately 12% MC for safe storage. Head rice yield (HRY), color, and other end−use qualities must be maintained during the drying process. Head rice yield is especially sensitive to the mode of drying since kernel fissures formed during the drying process can drastically reduce HRY. As such, HRY is commonly taken as an indicator to assess the success or failure of a rice drying system. A goal of the rice industry is to develop a rice drying process that can maximize throughput with minimal HRY reduction and efficient energy consumption. A key to attaining this goal is to understand the fundamental cause of fissure formation during the drying process.

The application of the $T_g$ concept to explain rice kernel fissure formation during the drying process was first proposed by Cnossen and Siebenmorgen (2000), although several researchers have recommended upper limits for rice drying air temperatures (Arora et al., 1973; Abe et al., 1992; Bonazzi et al., 1997). Cnossen and Siebenmorgen found that whether a rice kernel was above or below $T_g$ significantly affected several drying parameters, including drying rate and fissure initiation in the rice kernel.

If the rice kernel temperature is below $T_g$, then rice starch exists in a glassy state, the starch granules are compact, and water associated with the starch is relatively immobile. Drying at temperatures below $T_g$ yields a slow rate of moisture diffusion inside kernels, resulting in a slow drying rate and a longer duration to dry kernels to a targeted MC (Cnossen et al., 2002). If the drying temperature is above $T_g$, then the rice starch exists in a rubbery state, the starch macromolecules have greater free volume, and water in the starch is more mobile (Slade and Levine, 1991). Moisture can thus diffuse out of kernels much faster. To this end, the minimum drying air temperature for an effective drying process should, theoretically speaking, be chosen higher than $T_g$. However, the faster the moisture removal, the greater the MC gradient created because surface moisture is removed more quickly than that in the inner part of the kernel. Moisture content gradients within the kernel produce stress that can cause fissures to develop when the stress exceeds the tensile strength of the rice kernel (Kunze, 1979). Further, Cnossen and Siebenmorgen (2000) showed that fissures are

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produced when a rapid transition from the rubbery to glassy region is made if a sufficient MC gradient exists in the kernel during the transition. Inter−kernel MC gradients can be reduced by tempering, a process whereby kernels are placed in an environment in which drying is terminated while internal kernel moisture is allowed to migrate towards the surface of the kernel, thereby reducing differences in MC from the kernel center to the surface. Cnossen and Siebenmorgen found that tempering of rice kernels after a drying pass was required to maintain high milling quality if drying with air temperatures above $T_g$ and if sufficient MC gradients in kernels existed. Cnossen and Siebenmorgen (2000) concluded that drying air temperatures as high as 60°C could be used without reducing HRY as long as sufficient tempering at a temperature above $T_g$ was employed.

Several techniques can be used to measure the $T_g$ of polymeric materials, including differential scanning calorimetry (DSC) (Brandrup and Immergut, 1975), DMTA (Ferry, 1980), thermally stimulated current (Brandrup and Immergut, 1975), high−resolution nuclear magnetic resonance (NMR), and thermomechanical analysis (TMA) (Rabek, 1980; Perdon et al., 2000; Sun et al., 2002). Many researchers using these various methods have studied the glass transition and gelatinization behavior of starch/water systems. The most widely used method for this application is DSC. Most studies have focused on systems of starch in excess water (Stevens and Elton, 1971; Slade, 1984; Biliaderis et al., 1986; Yost and Hoseney, 1986; Slade and Levine, 1987, 1991; Marshall and Normand, 1991; Liu and Lelievre, 1991; Huang et al., 1994; Buera et al., 1998); only a few studies have been carried out in starch/water systems with a low MC (Slade, 1984; Slade and Levine, 1987, 1991; Zeleznak and Hoseney, 1987; Liu and Lelievre, 1991; Kalichevsky et al., 1992; Perdon et al., 2000). Slade (1984), Zeleznak and Hoseney (1987), and Slade and Levine (1987, 1991) showed that $T_g$ of wheat starch is extremely sensitive to MC. Zeleznak and Hoseney (1987) found that when MC was reduced from about 20% to 7%, $T_g$ of wheat starch increased from 30°C to 125°C.

Sun et al. (2002) determined that DSC was not sufficiently sensitive to accurately measure rice kernel $T_g$ due to rice kernels being a partially crystalline/partially amorphous polymer composite. In order to obtain a more accurate $T_g$ state diagram of rice kernels, DMTA was used in this study. In a DMTA test, the $T_g$ of a polymer can be determined from the changes in one of three different quantities: elastic storage modulus ($E'$), elastic loss modulus ($E''$), or their ratio ($E''/E'$). Each parameter can be used as a measure of the onset, midpoint, and end point of the glass transition. These are easily detected by DMTA for amorphous polymers, such as pre−gelatinized amylopectin and pre−gelatinized flour (Kalichevsky et al., 1992). This is due to the fact that $E'$ generally drops by several orders of magnitude and $E''$ and $E''/E'$ show sharp peaks around $T_g$. For ordered crystalline polymers, such as native starches and rice kernels, the changes in $E'$, $E''$, and $E''/E'$ are less drastic.

The specimen size requirements of a dynamic mechanical thermal analyzer pose difficulties for the application of DMTA in biological and food materials. Another problem involving DMTA measurement is the moisture loss of samples during testing (Pereira and Oliverira, 2000). Without protective means, samples can dry during measurement. Pereira and Oliverira (2000) tested different wrappings during DMTA tests of native wheat flour. It was found that when a sample was completely wrapped in aluminum foil, moisture loss was prevented. This method was used herein as part of the DMTA procedure for measuring the $T_g$ of brown rice kernels over a range of kernel MCs.

## MATERIALS AND METHODS

Two rice varieties were studied: Bengal (medium−grain) and Drew (long−grain). Both were harvested at the Rice Research and Extension Center near Stuttgart, Arkansas. Bengal was harvested at 19% MC in the autumn of 2000 (all MCs of rice kernels are expressed on a wet basis (w.b.) unless otherwise specified). Drew was harvested at 21.7% MC in the autumn of 1999. After harvest, rice was immediately transported to the University of Arkansas Rice Processing Lab, cleaned using a dockage tester (Carter−Day Dockage Co., Minneapolis, Minn.), and divided into different lots. Each rough rice lot was dried to different MC levels using 40°C and 17% relative humidity (RH) drying air. A temperature and RH control unit (Climate−Lab−AA, Parameter Generation and Control, Inc., Black Mountain, N.C.) maintained air conditions. Rice was dried in thin layers with this air to produce samples with MCs ranging from 7.6% to 21.7%. The MC of each bulk sample was measured by drying 15 g samples for 24 h in an air oven set at 130°C (Jindal and Siebenmorgen, 1987).

The procedure for preparing a rice kernel for DMTA testing consisted of first randomly selecting a rough rice kernel from one of the bulk samples. The hull was removed by hand to yield a brown rice kernel. In order to prepare the kernel to fit the sample requirements of the dynamic mechanical thermal analyzer and prevent moisture loss during the experiment, each brown rice kernel was individually wrapped with a thin layer of parafilm, a flexible, moisture−resistant film (laboratory film, American National Can, Chicago, Ill.) and then a thin layer of aluminum foil. The wrapped rice kernels were filed at both ends with fine sandpaper to produce 3 mm long rice cylinders with parallel ends. The heights of parafilm and aluminum foil were adjusted to be slightly shorter than the rice kernel to avoid any effect of wrapping material during a DMTA test. The lengths and diameters of each rice cylinder were measured before each DMTA test using a digital micrometer.

The transitions of brown rice kernels were determined using a dynamic mechanical thermal analyzer (DMTA−V, Rheometric Scientific, Piscataway, N.J.) over the temperature range of −40°C to 120°C. The samples were initially cooled to −40°C, held isothermally at −40°C for 5 min, and then heated from −40°C to 120°C at a rate of 3°C/min. A low frequency of 0.1592 Hz was used to minimize the frequency effect on $T_g$ (Ferry, 1980). DMTA tests were conducted in the parallel compression mode. It should be noted that other DMTA test modes, specifically tensile and three−point bending, were considered for these DMTA tests of rice kernels. However, due to kernel size and geometry constraints, only the compression mode was deemed reliable for reproducible $T_g$ measurements.

After the DMTA test, the parafilm and aluminum foil were removed and the brown rice kernel was weighed. The MC of the kernel was measured by placing the kernel in a porcelain spot plate and drying the sample for 24 h in an air oven set at

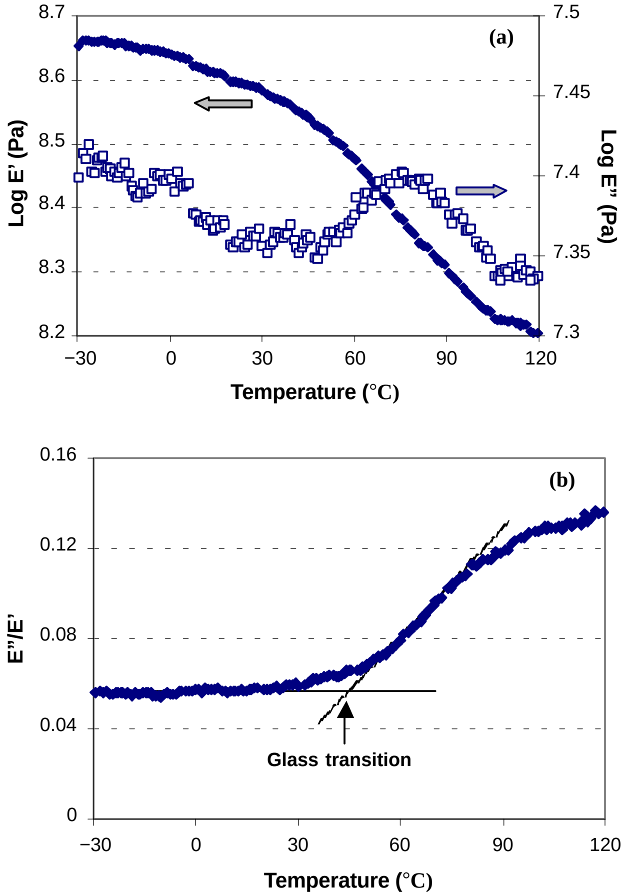


**Figure 1. Dynamic mechanical thermal analysis curves for a brown rice kernel (cv. Bengal) at 16.3% moisture content: (a) temperature dependence of the storage modulus (E′ ) and the loss modulus (E″ ), and (b) temperature dependence of E″ / E′ .**

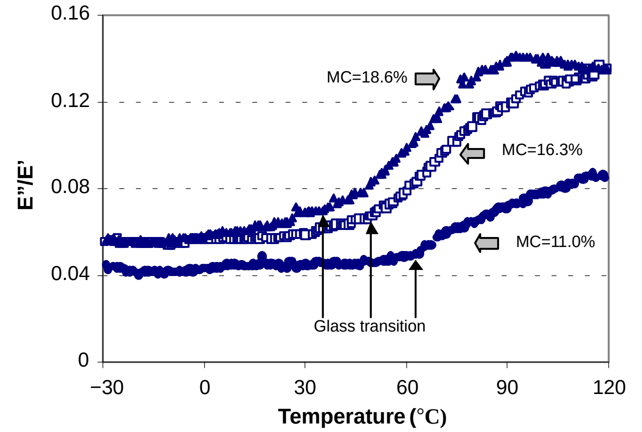


**Figure 2. The effect of moisture content (w.b.) on the spectra of E″ / E′ vs. temperature for brown rice kernels (cv. Bengal).**

130°C (Jindal and Siebenmorgen, 1987). This overall DMTA procedure was conducted on 70 kernels of each of the two rice varieties.

The $T_g$ values of rice variety Bengal were statistically compared to those of variety Drew. Since a linear relationship between $T_g$ and MC was observed in the tested MC range, the statistical analysis involved testing whether the regression lines of the $T_g$ values of Bengal and the $T_g$ values of Drew had the same slope and intercept. This was accomplished by means of linear regression with indicator variables (Neter et al., 1985):

$$T_g = b_0 + b_1 \times MC + b_2 X + b_3 MC \times X \qquad (1)$$

where $b_0$ to $b_3$ are regression constants, and $X$ is an indicator variable that assumes 0 for Bengal and 1 for Drew.

## Results and Discussion

### Tg Measurement

DMTA data were analyzed over a temperature range of −30°C to 120°C. Even though the DMTA measurement range extended to −40°C, the data from −40°C to −30°C were not used to avoid test specimen initialization at the start of a DMTA test. Figure 1a shows DMTA curves for a brown rice kernel at 16.3% MC. In the temperature range of −30°C to 120°C, there was a transition around 45°C, as indicated by the E′ and E″ responses to temperature. Initially, E′ decreased slowly as temperature increased, but decreased at a faster rate when the temperature was raised above 45°C. E″ initially decreased with an increase of temperature, but increased when the temperature was raised above 45°C and reached a peak around 80°C. However, changes in E′ and E″ were generally weak for most samples over the tested temperature range. Meanwhile, E″/E′ showed a much more definite transition. As shown in figure 1b, E″/E′ did not change with temperature below 30°C, but increased quickly thereafter. It is believed that the sharp increase in E″/E′ centered around 45°C was due to the glass transition of the rice kernel, based on this observation and those of others (Perdon et al., 2000; Sun et al., 2002).

The transition from a glassy to rubbery state occurs over a range of temperatures. Although the question of whether $T_g$ should be taken as the onset, the midpoint, or the end point of glass transition is a matter of discussion, it is reasoned to be more important to know the onset temperature of the glass transition process for rice drying applications. From figure 1, it is difficult to find and compare the peak and end point temperatures of glass transition of brown rice kernels from the E′, E″, and E″ / E′ vs. temperature curves, but it is clear that the onset temperatures of glass transition from E′, E″, and E″ / E′ are similar. Therefore, the onset temperature, as determined from the E″ / E′ vs. temperature relationship, was chosen to indicate the $T_g$ for brown rice kernels (fig. 1b).

### Effects of MC and Variety

The effect of MC on the E″ / E′ vs. temperature spectra for rice kernels is shown in figure 2. It is clear that the $T_g$ values of rice kernels are inversely dependent on MC, which is consistent with previous results (Perdon et al., 2000; Sun et al., 2002). This is more thoroughly illustrated in the plot of $T_g$ values versus kernel MCs in figure 3. This figure was generated by applying the approach described above in determining the onset temperature of the E″ / E′ vs. temperature curve for each brown rice kernel. In the tested MC range, the $T_g$ values of brown rice kernels were linearly related to MC.

The $T_g$ vs. MC relationship of Bengal (medium−grain variety) compared closely to that of Drew (long−grain variety); statistical tests (eq. 1, Neter et al., 1985) indicated that the data sets, approximated by a linear relationship

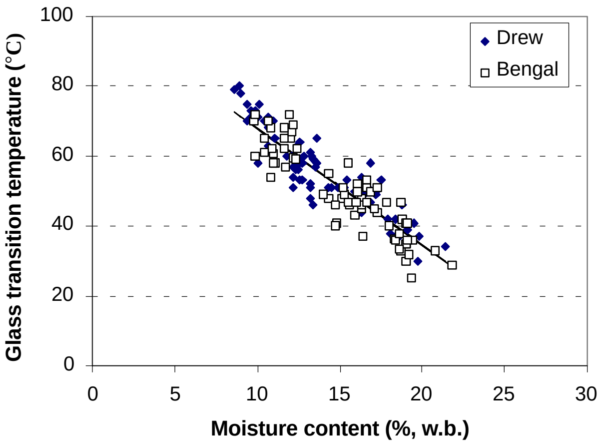


**Figure 3. Glass transition temperature vs. moisture content for Drew and Bengal brown rice kernels as determined by dynamic mechanical thermal analysis.**

between $T_g$ and MC, were not significantly different ($\alpha$ = 0.01). For rice variety Bengal, the $T_g$ vs. MC relationship was approximated by the following function:

$$Tg = 100.7 - 325 \times MC, \ R^2 = 0.82 \quad (2)$$

For rice variety Drew, the $T_g$ vs. MC relationship was:

$$Tg = 100.5 - 334 \times MC, \ R^2 = 0.81 \quad (3)$$

For the combined data, the $T_g$ vs. MC relation was:

$$Tg = 101.2 - 333 \times MC, \ R^2 = 0.82 \quad (4)$$

In equations 2 through 4, $T_g$ is in °C and MC is in decimal wet basis form. These results indicate that $T_g$ is independent of rice variety since there were no statistical differences in the slopes or intercepts of equations 2 and 3. This finding is consistent with that of Perdon et al. (2000), who observed no statistical differences in $T_g$ by TMA analysis between medium− and long−grain varieties. Statistical analyses also indicated that the average standard deviation of $T_g$ was 5.1°C.

### Comparison of Glass Transition Temperatures

The $T_g$ of rice kernels measured by DMTA in this study compared favorably with those measured by TMA (Sun et al., 2002) when MC was greater than 15% (fig. 4). However, the difference between the two methods was large when MC was lower, especially when less than 10%. Although it is difficult to conclude which method gives more accurate $T_g$ values of rice kernels, it is believed that the reliability of DMTA data is greater, especially at lower MCs. Since the TMA measurement of $T_g$ keys on a change in the thermal expansion coefficient as kernels incur a glass transition, and since this coefficient does not change dramatically at low MCs, increased variability and possible lowered accuracy in TMA measurement of $T_g$ could be expected with TMA.

A phase transition analyzer (PTA) is a closed−chamber capillary rheometer (Fujio et al., 1991; Hayashi et al., 1991; and Zhang et al., 1998). Several researchers have successfully used a PTA to measure the $T_g$ values and melting temperatures of different food polymer materials (Strahm, 1998; Strahm et al., 2000; Strahm and Plattner, 2000; Plattner et al., 2001). Plattner (2001) used a PTA to measure the $T_g$ of rice flour, and figure 5 illustrates the rice flour $T_g$ values from Plattner plotted along with the brown rice kernel $T_g$ values obtained in this study. While statistical tests were not conducted, figure 5 shows that the $T_g$ values of rice flour measured with a PTA were similar to the $T_g$ values of brown rice kernels measured by DMTA in this study.

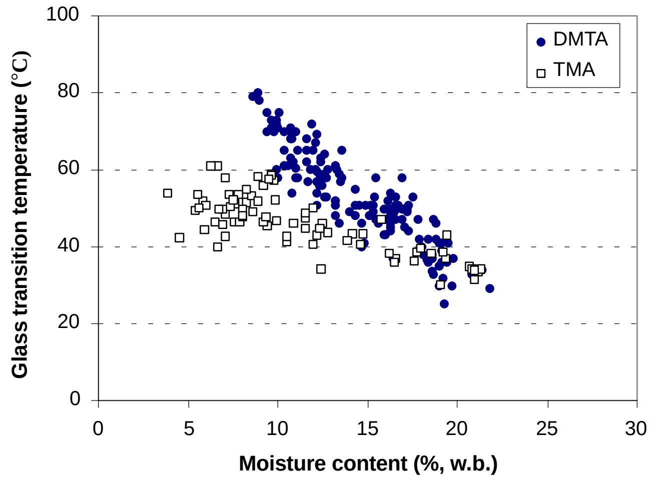


**Figure 4. Comparison of glass transition temperatures of brown rice kernels measured using dynamic mechanical thermal analysis (DMTA, data from fig. 3) and thermal mechanical analysis (TMA, Sun et al., 2002).**

## Conclusions

A protocol was developed to measure the $T_g$ of brown rice kernels using DMTA based on E″ / E′ vs. temperature curves. The following conclusions were made:

- The $T_g$ of brown rice kernels decreased with increasing MCs, following a linear relationship in the 7% to 22% MC range.
- There were no statistical effects ($\alpha$ = 0.01) due to rice variety, with a comparison made between medium− and long−grain varieties, on the $T_g$ of brown rice kernels.
- Glass transition temperatures measured by DMTA were in close agreement with those measured by TMA in an earlier study (Sun et al. 2002) when the kernel MC

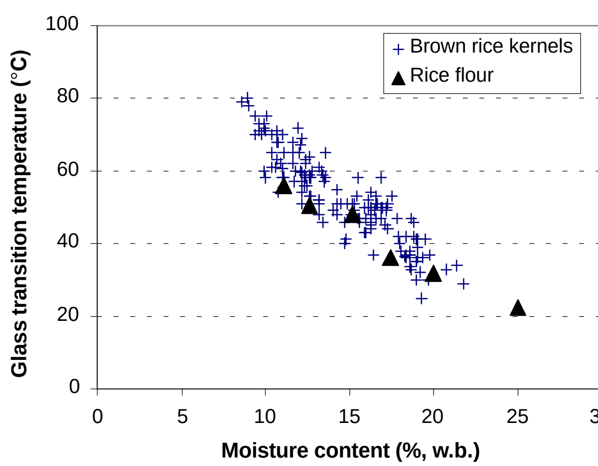


**Figure 5. Comparison of glass transition temperatures of brown rice kernels measured using dynamic mechanical thermal analysis (data from fig. 3) with those of rice flour as determined using a phase transition analyzer (Plattner, 2001).**

was greater than 15%. When the MC was less than 15%, $T_g$ values measured by DMTA were higher than those measured by TMA.

- Rice flour $T_g$ values reported by Plattner (2001) with a phase transition analyzer were similar to the $T_g$ values of brown rice kernels measured with DMTA in this study.


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