THE GLASS TRANSITION TEMPERATURE CONCEPT IN RICE DRYING AND TEMPERING: EFFECT ON MILLING QUALITY

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ABSTRACT. *A true understanding of rice kernel fissuring and breakage, as a result of drying and tempering, must include both engineering and cereal science principles. Particular emphasis must be placed on the change of states of starch occurring at the glass transition temperature (T_g). This transition from a glassy to rubbery state, or vice versa, has been identified to play an important role in rice fissuring and breakage. A hypothesis has been developed explaining rice kernel fissuring during drying and tempering. The objectives of this research were to determine the effect of the T_g during rice drying and tempering on milling quality. Additionally, the minimum tempering time required for various drying conditions, to optimize milling quality, was determined. Rice was dried under three conditions, two with a drying air temperature above T_g and one below T_g, for four durations and then tempered for 0 to 240 min. The experimental procedure was designed to directly test the T_g hypothesis by cooling rice to a temperature below the T_g after each tempering duration. Results for both medium-grain rice and long-grain rice at 19.6 to 23.7% harvest moisture content (MC)* show that 5 to 6 percentage points MC can be removed per drying pass without damaging the rice kernel, as long as sufficient tempering is allowed. Required tempering durations were shorter for long-grain rice as compared to medium-grain.*

Keywords. *Rice, Glass transition temperature, Drying, Tempering, Head rice yield.*

Commercial dryers use multi-pass procedures to remove moisture from freshly harvested rice. Between drying passes, the rice is held in bins for a certain period of time to allow moisture content (MC) gradients within kernels, created during drying, to subside. This holding process is referred to as tempering. Tempering decreases MC gradients by allowing moisture to migrate from the core to the outer layers of the kernel. The MC gradients cause differential stress inside the kernel, which, if sufficiently large, causes the kernel to fissure (Kunze, 1979). Moisture migration during tempering also improves the energy utilization in subsequent drying passes (Steffe and Singh, 1980).

Fissured kernels may break during the milling process and will thus reduce head rice yield (HRY). HRY is the current standard in the rice industry to measure rice milling quality and is defined as the weight percentage of rough rice that remains as head rice (kernels that are at least three-fourths of the original kernel length) after complete milling.

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* All moisture contents are expressed on a wet basis.

In commercial rice drying, tempering practices vary widely. Tempering times between 6 and 24 h are used in the U.S. (Mossman, 1986; Steffe and Singh, 1980). By determining the minimum tempering time required to sufficiently reduce kernel MC gradients, the drying process can be optimized.

TEMPERING RESEARCH

Many researchers have studied the tempering process in rice drying. The emphasis in most of the studies was on the effect of tempering duration on drying performance (i.e., drying rate and energy utilization). Few researchers (Beeny and Chin, 1970; Cnossen et al., 1998; Steffe et al., 1979; Wasserman et al., 1964), however, have studied the effect of tempering on kernel quality.

A theoretical model, based on initial MC and drying parameters, to predict tempering time for rough rice was developed by Steffe and Singh (1980). According to the model, tempering, which was based on the relative humidity (RH) response of the inter-kernel air, was 95% complete in less than 2 h and fully complete in less than 5 h, when using a 35°C drying air temperature. This work was conducted using a short-grain rice variety. Steffe et al. (1979) concluded that a 35 min tempering duration was sufficient to equalize the MC gradient in the rice kernel, after drying high MC medium-grain rice for 20 min using 38°C air. Three hours tempering time was sufficient after drying for 35 min using 38°C air or after drying for 20 min using 50°C air. Tempering did improve head rice yield (HRY), but HRYs were equal for any length of tempering considered.

High tempering temperatures have been shown to be effective in maintaining high HRYs and decreasing tempering duration. Beeny and Chin (1970) dried rice with an initial MC of 24% using 54.4°C air and found an increasing HRY with increasing tempering duration up to 5 h. Cnossen et al. (1998) showed increasing HRYs for tempering durations up to 150 min when drying medium-grain rice with 60°C air and tempering at this drying temperature. Wasserman et al. (1964) showed increasing HRY and decreasing tempering duration with increasing tempering temperature for a short-grain rice variety dried using 43.3°C air. HRY was 2% higher for rice tempered warm (40.6°C) compared to rice tempered cold (23.8°C). Samples tempered at 40.6°C required 4 h of tempering while samples tempered at 23.8°C required 6 h. Steffe and Singh (1980) found similar trends of decreasing tempering duration with increasing temperature. Nguyen and Kunze (1984) concluded that a storage temperature of 45°C after drying reduced the number of fissured grains by an average of 25.5% compared to a storage temperature of 10°C, when drying Brazos rice at 60°C.

Rice Fissure Formation

Drying and tempering processes affect the rice kernel structure, particularly fissure formation. Understanding these effects is important in controlling and optimizing drying and tempering conditions. Theories on fissure formation are based on the response of rice kernels when subjected to tensile and compressive stresses due to the existence of an MC gradient within the kernel (Kunze and Choudhury, 1972).

Several researchers (Ban, 1971; Kunze, 1979; Nguyen and Kunze, 1984; Sharma and Kunze, 1982) observed that fissures did not occur until after drying had ceased. Few solid whole rough rice kernels will fissure during drying (Sharma and Kunze, 1982). Severe drying conditions can increase the number of kernels that fissure after drying. Since most kernels are not fissured immediately after drying, Sharma and Kunze (1982) suggested that there is a possibility that ". . . some post-drying treatment or procedure can be developed that will prevent the subsequent fissures". Research (Wasserman et al., 1964; Nguyen and Kunze, 1984; Cnossen et al., 1998) has shown that proper tempering using high tempering temperatures can minimize the potential for kernel damage from severe drying conditions.

Material Property Considerations

Perdon and Siebenmorgen (1999) and Siebenmorgen and Perdon (1999) suggested that a complete and fundamental understanding of the response of kernels to various drying and tempering environments must include considerations of material properties at the temperature and MC of various sections of the kernel. A true understanding of kernel drying and tempering requires an integration of engineering principles, such as heat and mass transfer theory, and cereal science principles dealing with material properties (Siebenmorgen, 1998).

Polymer science has been applied in studying the effect of temperature and MC changes during processing on food components such as starch and protein (Slade and Levine, 1991, 1995; Perdon, 1999). Starch, the main constituent of rice, is considered a polymer of glucose whose thermal and material properties change depending on the temperature and MC gradients generated during processing (Slade and Levine, 1991, 1995). The change of state of starch, as it goes through a glass transition, has been identified (Perdon, 1999) to play an important role in rice drying and tempering in terms of kernel fissuring potential. Perdon and Siebenmorgen (1999) concluded that this state transition occurs in the temperature range typically encountered during rice drying, and would affect the material properties in a rice kernel.

At a temperature and MC below the glass transition temperature (T_g), starch exists as a 'glassy' material, with low expansion coefficients, specific volume, and diffusivity. As the kernel temperature passes through T_g, the starch goes from a "glassy" into a "rubbery" state. Above the T_g, starch exists as a rubbery material with higher expansion coefficients, specific volume, and diffusivity (Slade and Levine, 1991, 1995; White and Cakebread, 1966). Figure 1 shows a T_g relationship for rice variety Bengal measured by Perdon (1999), identifying the glassy region and the rubbery region. Figure 1 indicates that the T_g is inversely related to MC, i.e., as MC increases, T_g decreases. Perdon (1999) showed that the physical properties of a rice kernel changed dramatically as the kernel temperature passed through T_g.

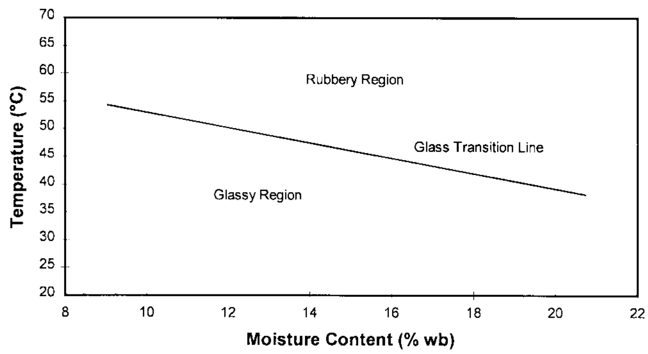

Figure 1–Glass transition relationship for variety Bengal brown rice indicating the glassy and the rubbery region (Perdon, 1999).

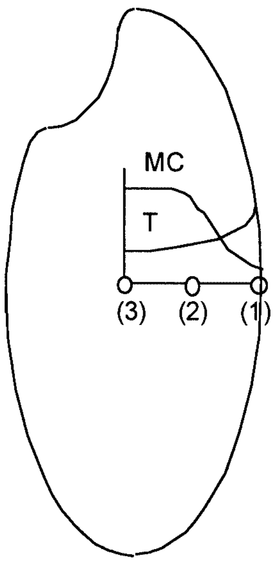

Figure 2–Hypothetical temperature (T) and moisture content (MC) distribution within a rice kernel during drying plotted onto a rice kernel. Points 1, 2, and 3 on the x-axis of the plot correspond respectively to the surface, mid-point between the surface and the center, and the center of the kernel.

Figure 2 depicts hypothetical temperature and MC gradients created within a rice kernel during drying plotted onto a rice kernel. During drying, the MC of the outer layers of the kernel is lower than the MC of the center, causing the surface layer T_g to be higher. Therefore, a T_g gradient develops within the kernel with the outer endosperm having a higher T_g than the center. During tempering or during cooling, depending on the temperature that the kernel is exposed to, the outer layer may be glassy, while the center is still in a rubbery state. Consequently, the different layers would be characterized by different magnitudes of material properties.

The temperatures and MCs inside a kernel, after a certain period of drying, are superimposed onto a state diagram for a brown rice kernel in figure 3. The points surface, mid-point between the surface and the center, and center correspond to the points 1, 2, and 3, respectively, in figure 2. The temperature in the entire kernel increases fairly rapidly to the temperature of the drying air, and the outer layers of the kernel lose moisture. Figure 4 shows the same rice kernel after a longer period of drying, indicating a large MC gradient inside the kernel.

During the tempering stage, following a drying pass, moisture will migrate from the center to the surface of the kernel (Kunze, 1979) and consequently the MC gradient will decrease. If the tempering temperature is below the T_g of the rice, the kernel will again go through a glass

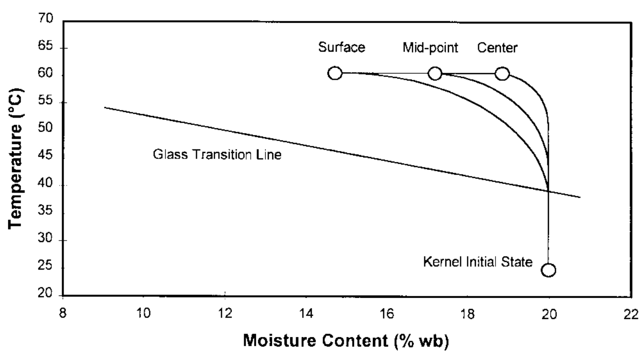

Figure 3–Hypothetical temperatures and moisture contents (MCs) within a brown rice kernel after removing a small amount of moisture (2 to 3 percentage points overall MC reduction), thus producing an MC gradient from the surface to the center of the kernel.

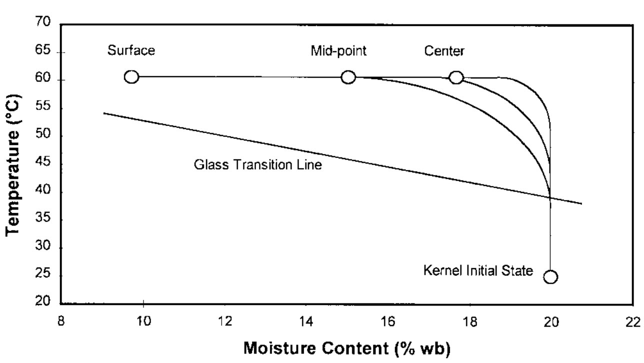

Figure 4–Hypothetical temperatures and moisture contents (MCs) within a brown rice kernel after a longer period of drying than that depicted in figure 3, removing a large amount of moisture (4 to 5 percentage points overall MC reduction).

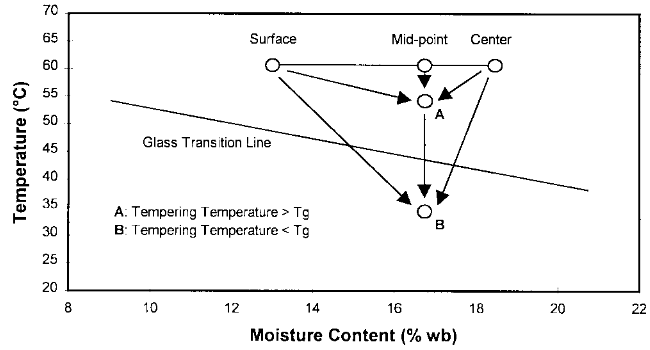

Figure 5–Hypothetical response of the various sections of a brown rice kernel during tempering for two tempering scenarios.

transition and become glassy as the kernel temperature decreases. It is hypothesized that if the tempering environment is one that produces a change of state of the starch, differential stresses within the kernel, resulting from the temperature and MC gradients, could cause kernel fissuring. This scenario is depicted by situation ‘B’ in figure 5 in which the tempering temperature is less than the T_g of the rice and the surface, mid-point, and center of the kernel cross the T_g line at different MCs. Our hypothesis would indicate that this scenario would create kernel fissuring, with resultant HRY reduction, if there was a sufficient MC gradient inside the kernel. This hypothesis would agree with the observations (Ban, 1971; Kunze, 1979; Nguyen and Kunze, 1984; Sharma and Kunze, 1982) that rice kernels fissure after the drying process has ceased.

Most of the previous research on rice tempering was directed to short-grain varieties, using gentle drying conditions with temperatures below the T_g of the rice. Also, limited research has been done on the influence of high tempering temperatures on rice quality. Steffe and Singh (1980) and Wasserman et al. (1964) concluded that higher tempering temperatures would decrease the required tempering duration. Consequently, there is a need for further research on the influence of high drying and tempering temperatures on the milling quality of medium- and long-grain rice varieties.

The following objectives were formulated for this study:

1. Investigate the effects of glass transition and high drying and tempering temperatures on HRY reduction during tempering.
2. Investigate a minimum tempering duration required between drying passes, while maintaining high HRYs, for both medium- and long-grain rice varieties.

Experimental Design

Rice varieties Bengal (medium-grain) and Cypress (long-grain) at different harvest moisture contents (HMCs), high (20 to 21%) and low (18 to 19%), were harvested from University of Arkansas Research and Extension Centers at Stuttgart and Keiser, Arkansas, in 1998. Immediately after harvest, the rice was cleaned with a Carter-Day Dockage tester (Carter-Day Co., Minneapolis, Minn.).

The experimental design consisted of drying experiments with the drying air and tempering

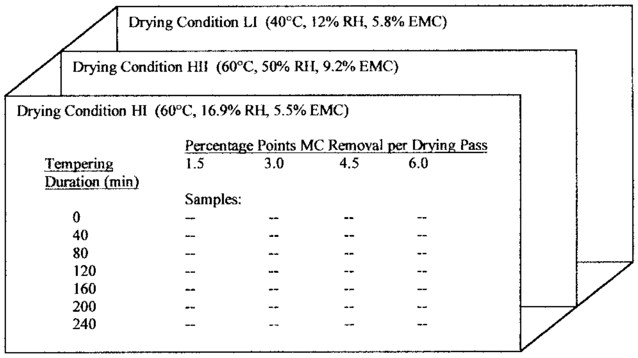

Figure 6–Experimental design for the drying and tempering experiments. Layout represents the sampling routine for each harvest location/variety/harvest moisture content lot.

temperatures above and below the T_g of the rice (fig. 1), to provide insight into the T_g hypothesis of fissuring. Figure 6 illustrates the experimental design. The rice lots were dried at three drying air conditions: condition HI (60°C, 16.9% RH), resulting in 5.5% equilibrium moisture content (EMC) as predicted by the Chung equation (ASAE, 1998); condition HII (60°C, 50% RH), resulting in 9.2% EMC; and condition LI (40°C, 12% RH), resulting in 5.8% EMC. Thin layer (3 to 5 mm) drying was achieved by using perforated trays in drying chambers in which the drying air conditions were controlled by PG&C 8.5 m^3min^{-1} temperature and RH controllers (Parameter Generation & Control Inc., Black Mountain, N.C.). For each drying condition, samples were dried for four different durations aiming at removing 1.5, 3, 4.5, and 6 percentage points MC. The different magnitudes of MC gradient, created by these different drying durations, provided information on the maximum allowable MC gradient inside a rice kernel before it fissured. Two drying replications were performed for each drying condition/duration combination. Weight loss of the rice was monitored during drying to terminate drying as soon as the desired MC reduction was achieved. After each drying run, the actual MC loss of the sample was measured by drying duplicate 15-g samples for 24 h in an oven set at 130°C (Jindal and Siebenmorgen, 1987).

Immediately after a drying run, the rice batch was split into seven sub-samples, which were tempered for varying durations ranging from 0 to 4 h in 40-min increments in an oven set at the temperature of the dryer. The varying tempering durations created varying levels of MC equilibration in the kernels, i.e., a tempering duration of zero minutes resulted in maximum MC gradient while extended tempering resulted in reduced gradients. Tempering of samples was performed in sealed bags. After tempering, the samples were taken out of the sealed bags and placed into an EMC chamber maintained at 21°C and 55% RH to gently dry to 12.5% MC. This 21°C temperature is well below the T_g of the rice at typical MCs. Thus, the kernels were forced to undergo a state transition with varying levels of MC gradients due to the varying tempering durations.

Prior to milling, the samples were held in storage at 4°C for three months. A Satake laboratory huller was used to dehull the rice. Samples were subsequently milled for 30 s in a McGill no. 2 mill, resulting in a degree of milling of 80 to 90 as measured with a Satake MM-1B milling meter. The mass of head rice was determined using a FOSS Graincheck 310 image analyzer (Foss North America, Minneapolis, Minn.) and the HRY calculated as the mass fraction of rough rice remaining as head rice.

As a means of control, two samples of rice from each harvest lot were gently dried in the EMC chamber from HMC to 12.5% MC, resulting in minimal breakage and consequently a high HRY. The HRYs of the samples having received different treatments were then compared against the HRY of the control sample.

Additional drying tests were conducted to measure the RH of the inter-kernel air during tempering. As tempering progresses, the RH of the inter-kernel air reaches a steady state, indicating that there is no additional moisture transfer from the kernel to the surrounding air. The RH of the inter-kernel air has been used as an indicator for determining minimum tempering durations (Steffe and Singh, 1980) and was used in this study as additional information to determine MC gradient relaxation inside the rice kernels. Rice samples were dried under the two drying conditions with a drying air temperature above T_g (conditions HI and HII). Under each condition, 3, 4.5, and 6 percentage points MC were removed. After drying, the samples were placed in a sealed flask and tempered at the same temperature as the dryer for 150 min. As a means of measurement, a temperature and RH probe was placed inside the sealed flask. The RH was then plotted against tempering duration.

Results and Discussion

The HMC of each lot is shown in table 1. The overall HMCs were 1 to 2 % higher, due to weather circumstances, than the target HMCs described in the experimental design. Before drying was started, the HMC was measured with a Motomco 919A moisture meter (Dickey-john, Auburn, Ill.). An accurate HMC, determined by the 24-h oven procedure (Jindal and Siebenmorgen, 1987), was not available when the drying runs were started since drying was started as soon as possible after harvesting. Therefore, the actual percentage points MC removed during each drying run, measured with the oven method, deviated from the target MCs calculated with the initial measure of HMC with the moisture meter. This also affected the subsequent weight loss procedure for several drying runs.

Relative Humidity Measurements

Numerous EMC studies on grains have shown that there is a fixed relationship between the moisture concentration of the kernel and the RH of the air surrounding the kernel (Fan et al., 1999; Steffe and Singh, 1980). Therefore, the RH of the inter-kernel air can by used as an indicator of MC gradient relaxation. To quantify the degree of MC

Table 1. Harvest moisture contents (HMCs) of the rice lots

Lot	Variety	HMC (%)	Harvest Location
2	Cypress	20.0	Stuttgart, Ark.
3	Cypress	20.6	Stuttgart, Ark.
4	Bengal	21.3	Stuttgart, Ark.
5	Bengal	19.6	Stuttgart, Ark.
6	Cypress	23.7	Keiser, Ark.
7	Bengal	21.3	Keiser, Ark.
8	Cypress	20.9	Keiser, Ark.

equalization in a rice kernel, Steffe and Singh (1980) defined a tempering index as:

$$I_{RH} = \frac{RH(t) - RH(t=0)}{RH(t=\infty) - RH(t=0)} \qquad (1)$$

where the RH at time equals zero is the RH at the start of tempering and RH at time infinity is the maximum RH of the inter-kernel air obtained during tempering. The equation was used to calculate the degree of tempering. To quantify minimum tempering durations for the different drying durations, the time at which the I_{RH} was equal to 0.95 was calculated. This was defined as the 95% point at which tempering was 95% complete.

Figure 7 shows the RH of the inter-kernel air during tempering for variety Cypress rice. As can be seen in figure 7, the difference in time for the curve to reach final RH was small among the three drying durations. Tempering was 95% complete in 31 min for the rice that was dried for a duration resulting in 3 percentage points MC removal, while for the durations resulting in 4.5 and 6 percentage points MC, tempering was 95% complete in 43 and 42 min, respectively. Variety Bengal also showed a small difference in time to reach the 95% point between drying durations (25, 37, and 51 min, respectively, when removing 3, 4.5, and 6 percentage points MC under the 60°C, 16.9% RH drying condition for harvest lot 4).

The asymptotic RH of the inter-kernel air decreased with increasing drying duration. Entering the measured final RH of the inter-kernel air after 150 min of tempering into the Chung equation for calculating EMC (ASAE, 1998) did not agree with the MC of these samples obtained with the oven method (Jindal and Siebenmorgen, 1987). The calculated EMC was 2 to 3 percentage points higher than the actual MC obtained with the oven method. This difference could be due to the inability of the Chung equation to predict accurate EMCs at high temperatures or varietal differences not accounted for in the equation. Fan et al. (1999) also found varietal differences in EMCs at a given RH in the temperature range of 4 to 38°C.

DRYING ABOVE T_g: HRY RESULTS

According to our hypothesis, if rice is tempered above the T_g line sufficiently long enough to reduce MC gradients, a state transition will not cause HRY reduction. Insufficient MC gradient reduction before a state transition will produce fissures and consequent HRY reduction.

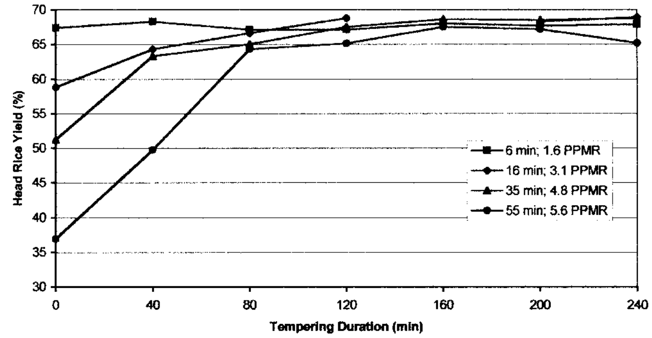

Figure 8–Head rice yield (HRY) versus tempering duration for variety Bengal rice dried for four durations (PPMR is percentage points moisture content removed) with 60°C, 16.9% relative humidity drying air (condition HI) (Harvest lot 7, table 1). The HRY of the control sample was 67.9%.

Figure 8 shows the HRYs of Bengal (harvest lot 7, table 1) samples for different tempering durations, after different percentage points MC were removed under the 60°C, 16.9% RH drying condition (HI). From figure 8, removing 1.6 percentage points MC caused little damage to the rice, and tempering had no effect. This indicates that for this drying duration, sufficient MC gradients were not produced during drying to create fissures when the rice was placed in the 21°C environment and forced to undergo a state transition. However, when removing up to 4 8/10 percentage points MC without tempering (0 min tempering duration), a dramatically lower HRY was observed. Furthermore, the samples that were tempered for 40 min showed a significant increase in HRY, and the samples tempered for 120 min did not show any HRY reduction compared to the control sample. When removing up to 5 6/10 percentage points MC, 160 min tempering was necessary to achieve a HRY equivalent to that of the control. This indicates that, after these tempering durations, the MC gradients were sufficiently reduced and did not lead to fissuring.

Up to 6.5 percentage points MC were removed for harvest lot 4, dried under the 60°C, 50% RH drying condition (HII), as shown in figure 9. The samples that had

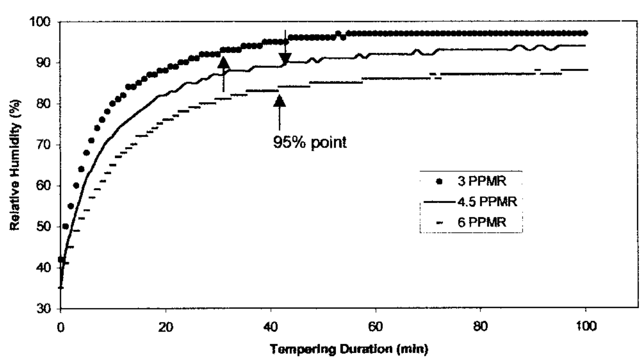

Figure 7–Relative humidity (RH) of the inter-kernel air during tempering of Cypress rice at 60°C, after three drying durations (PPMR is percentage points moisture content removed) using 60°C, 16.9% RH drying air (condition HI). Arrows show the time at which tempering was 95% complete (Harvest lot 8, table 1).

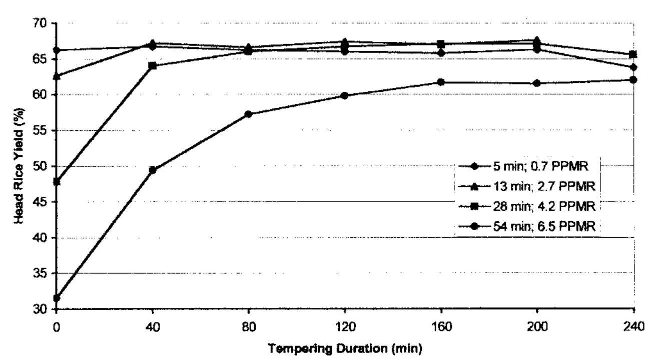

Figure 9–Head rice yield (HRY) versus tempering duration for variety Bengal rice dried for four durations (PPMR is percentage points moisture content removed) with 60°C, 50% relative humidity drying air (condition HII) (Harvest lot 4, table 1). The HRY of the control sample was 65.7%.

6.5 percentage points MC removed showed significant irreversible structural damage; even after 240 min tempering the HRY was 5 7/10 percentage points lower than the HRY of the control sample. When looking at the drying durations that did not cause irreversible structural damage (up to 4 2/10 percentage points MC removal), the samples dried under the high RH air condition had HRYs closer to the HRY of the control samples for the 40 and 80-min tempering durations compared to the samples in figure 8. This indicates that these samples tempered faster compared to samples dried under the 60°C, 16.9% RH drying condition. This trend was consistent across the all the Bengal lots.

Figure 10 represents the HRY results for variety Cypress (harvest lot 3, table 1) when dried under the 60°C, 16.9% RH drying condition. Required tempering durations, to maintain a high HRY, were significantly lower for the long-grain Cypress rice compared to that of the medium-grain Bengal rice. Tempering durations of 40 min were sufficient when removing up to 4 8/10 percentage points MC, and 80 min of tempering was needed to reduce the MC gradients when removing up to 6.5 percentage points MC. Due to a thinner kernel, the magnitude of the MC gradient in a long-grain rice kernel could be smaller compared to that of a medium-grain, resulting in a faster equilibration time. The differences in tempering duration between rice dried under the low RH drying condition and rice dried under the high RH drying condition that were observed with the medium-grain variety were not observed with the long-grain variety.

HMC had a significant influence on tempering duration; the lower the HMC, the longer the required tempering duration. Cypress harvested at 23.7% MC (harvest lot 6) required 40 min of tempering when removing 6 2/10 percentage points MC, while Cypress harvested at 20.6% MC (harvest lot 3) required 80 min of tempering when removing 6.5 percentage points MC, when dried under the 60°C, 16.9% RH drying condition. Similar trends were observed for Bengal rice.

Drying Below T_g: HRY Results

Due to a slower drying process, resulting in less severe MC gradients, and the fact that a state transition did not occur when cooling, tempering had little effect on the HRY

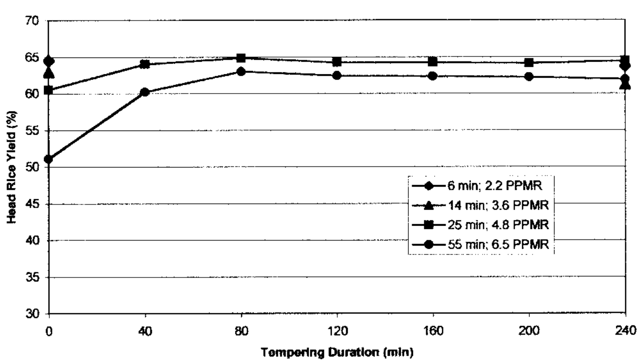

Figure 10–Head rice yield (HRY) versus tempering duration for variety Cypress rice dried for four durations (PPMR is percentage points moisture content removed) with 60°C, 16.9% relative humidity drying air (condition HI). For the 2.2 and 3.6 PPMR durations only the 0 and 240 min tempering durations are depicted (Harvest lot 3, table 1). The HRY of the control sample was 64.5%.

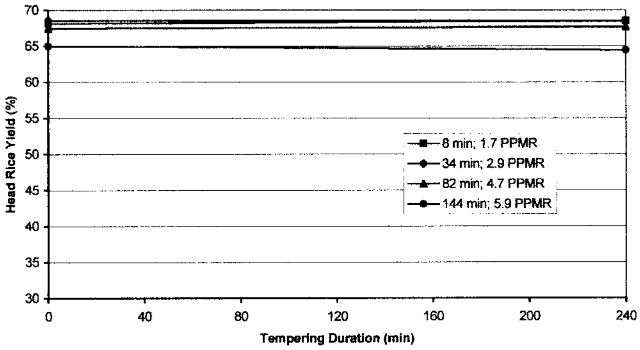

Figure 11–Head rice yield (HRY) versus tempering duration for variety Bengal rice dried for four durations (PPMR is percentage points moisture content removed) with 40°C, 12% relative humidity drying air (condition LI) (Harvest lot 7, table 1). The HRY of the control sample was 67.9%.

for both varieties when rice was dried using a drying air temperature below the T_g. The slower drying process did not cause a sufficient MC gradient inside the kernel to cause significant fissuring and subsequent breakage. Figure 11 shows the HRY versus tempering duration for Bengal rice dried under the 40°C, 12% RH drying condition. Figure 11 shows a small decrease in HRY with increasing amounts of MC removal. Variety Cypress showed little HRY reduction for prolonged drying durations. HMC did not have a significant effect on HRY results when using drying air temperatures less than T_g.

Conclusions

The following conclusions were drawn from this study:

1. Drying air temperatures as high as 60°C can be used without reducing the HRY as long as sufficient tempering, at a temperature above the T_g of the rice, is allowed.
2. The RH measurements showed that a much shorter tempering duration is required to decrease the MC gradient inside a rice kernel than the HRY results did.
3. Tempering rice immediately after drying had a significant effect on HRY when the drying air conditions produced sufficient MC gradients inside the kernel and produced a state transition that placed the kernel, or parts of the kernel, into the rubbery region. For both variety Bengal and variety Cypress rice and for both drying conditions HI and HII, 5 to 6 percentage points MC could be removed in a single drying pass without damaging the kernel, if the rice was tempered at 60°C before cooling to 21°C. For Bengal, 160 min of tempering was sufficient and for Cypress a tempering duration of 80 min was sufficient, based on achieving HRYs equivalent to that of the control samples.

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