



GLASS TRANSITION AND TECHNOLOGICAL PROPERTIES IN DRYING AND TEMPERING OF BROWN RICE GRAINS

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Keywords: drying methods; temperature; relative humidity; air flow rate.

Abstract

The objective of this study was to determine the glass transition curve and to evaluate the influence of drying air temperature, grain layer thickness and implementation of the tempering step (with thermal and hydraulic insulation), on rice grains, long-thin class. The experiment was carried out using a reduced-scale silo-dryer, set to keep the average drying air temperature at 30, 55 and 80 °C. The thickness of the grain layer was 0.45 m, divided into three layers (lower, middle and upper) of equal dimensions. Immediately after drying, the grains were subjected to tempering for 0 (no tempering), 120 and 240 min. It was analyzed Determination of the initial and final moisture content of the grains, Technological properties, Determination of glass transition curve and Cooking properties. It was concluded through this study that grain layer thickness interfered with drying time, with a difference of up to 3 h more for the upper layer in comparison to the lower one. Implementing the tempering step has preserved the quality of rice grains, as the drying with average air temperature of 55 °C (40 °C in the grains) and tempering for 240 min led to results equivalent to those obtained in the drying with air temperature of 30 °C (control).

TRANSIÇÃO VÍTEA E PROPRIEDADES TECNOLÓGICAS NA SECAGEM E TEMPERAGEM DE GRÃOS DE ARROZ EM CASCA

Palavras-chave: métodos de secagem; temperatura; umidade relativa; fluxo de ar.

Resumo

O objetivo deste estudo foi determinar a curva de transição vítrea e avaliar a influência da temperatura do ar da secagem, da espessura da camada de grãos e da implantação da etapa de temperagem (com isolamento térmico e hídrico), em grãos de arroz, classe longo-fino. O experimento foi realizado com uso de equipamento silo-secador de escala reduzida, ajustado para manter a temperatura média do ar de secagem em 30, 55 e 80 °C. A espessura máxima da camada de grãos, durante as secagens, foi de 0,45 m, dividida em três camadas (inferior, média e superior) de dimensões iguais. Imediatamente após a secagem, os grãos foram submetidos à temperagem durante 0 (sem temperagem), 120 e 240 min. Foram realizados as análises de umidade, propriedades tecnológicas, curva de transição vítrea e parâmetros de cozimento. Concluiu-se através deste estudo que a espessura da camada de grãos interferiu no tempo de secagem, com uma diferença de até 3 h a mais, da camada superior em relação à inferior. A implementação da etapa de temperagem preservou a qualidade dos grãos de arroz, onde a secagem com temperatura média do ar de 55 °C (40 °C nos grãos) e temperagem com duração de 240 min apresentou resultados equivalentes à secagem com temperatura do ar de 30 °C (de controle).

INTRODUCTION

The drying process is essential for the conservation of rice grains, preventing the incidence of fungi and insects (GUENHA et al., 2014). In rice drying, the physical integrity of the grains depends on extrinsic factors, related to both the drying air and the dryer, and on intrinsic factors that vary depending on the cultivar (TIMM et al., 2020). One intrinsic factor of great importance in the drying of grains, which concerns the possibility of change in the physical state of some of their constituents, is called the glass transition temperature (T_g).

The starch, main constituent of rice grains, when subjected to a drying condition below the glass transition temperature, remains in a vitreous state, with low coefficients of expansion, specific volume and diffusivity, and the water contained within the grain has low mobility, increasing its drying time (PERDON et al., 2000). When the grain is subjected to a drying condition above its glass transition temperature, the starch changes to an amorphous state, showing high coefficients of expansion, specific volume and diffusivity and called rubbery state. Therefore, such glass transition, which may occur during drying and/or shortly after it, during the period of natural cooling of the grains (tempering), is directly related to the increase of moisture, temperature and tension gradients within the grains, which can cause cracks, increase the incidence of broken grains and substantially reduce head rice yield (MUKHOPADHYAY and SIEBENMORGEN, 2018). Currently, many rice drying systems do not yet include a proper tempering in their projects, but studies indicate that, immediately after drying of rice with hot air, it is necessary to apply an adequate tempering to reduce the tensions generated within the grain, preserving its physical integrity, quality and value (MUKHOPADHYAY et al., 2019).

The objective of this study was to determine the glass transition curve and to evaluate the influence of drying air temperature, grain layer thickness and implementation of the tempering step (with thermal and hydraulic insulation). For this, kinetic drying curves were evaluated based on the glass transition curve of the studied rice, and the effects of drying and

tempering of the rough rice were investigated based on the physical, technological and cooking properties of grains processed by the conventional system (white rice polished).

MATERIAL AND METHODS

Material

Grains of the rice cultivar 'Guri Inta' (long-thin class), produced under irrigated system in southern Brazil, were harvested with 26% moisture content (dry basis) and cleaned in a cleaning machine (Intecnia brand, Sintel model, Brazil). The rice samples were placed in bags and immediately transported to the Laboratory of Post-harvest, Industrialization and Quality of Grains of the Federal University of Pelotas, where the drying procedure and the study were conducted.

Experimental procedure

The grains were dried, until they reach 13% average moisture content (dry basis), in stationary silo-dryer, system widely used by producers to dry and store grains. This silo-dryer is composed of a centrifugal fan and a set of electric resistances connected to an electronic temperature control system. The drying chamber of the silo-dryer consists of a metal cylinder with 0.25 m internal diameter and 1.00 m height. The dryer ventilation system was adjusted through the suction inlet, to provide specific air flow in the order of $14 \text{ m}^3 \text{ air min}^{-1} \text{ t}^{-1}$, a value close to those of industrial hot air dryers (LANG et al., 2018). In order to avoid the direct contact of the hot drying air with the grain sample, a bag of permeable fabric with 2.0 kg of previously dried grains (with 12% moisture content, dry basis), replaced in every drying and repetition, was maintained immediately above the perforated plate (false bottom) of the drying chamber during all treatments. In each drying/treatment, three (3) permeable fabric bags with 4 kg of rough rice grains, representing a 0.15-m-thick layer for each bag, resulting in a total grain layer (bed) thickness of 0.45 m and total mass of 12 kg. The drying operation was adjusted to keep the air temperature at $30, 55$ and $80 \pm 1 \text{ }^\circ\text{C}$. During these drying procedures, the grain mass temperature was evaluated by wireless (radio frequency transmission) thermal sensor inserted into the center of the respective

layer and the removal of grain moisture content was calculated by the difference between the initial and final masses (periodically measured by the load cell of a balance with 0.1 g accuracy) in each layer (bottom with thickness from 0.00 to 0.15 m, intermediate with thickness from 0.15 to 0.30 m and top with thickness from 0.30 to 0.45 m). The tempering step was performed by placing the permeable fabric bags with dry grain samples into high-density polypropylene bags hermetically sealed for insulation against moisture and then placing these bags into thermal expanded polystyrene boxes with 3.65-cm-thick wall, for thermal insulation, for tempering periods of 120 or 240 min, plus a condition in which the sample was not subjected to the tempering step, but to a natural balance with the environment, without thermal and moisture insulation, represented here by tempering without tempering. There were three replicates for each tempering duration. Subsequently, after 4 h of rest, the samples were stored at a temperature of 20 °C for 72 h, to perform the analysis.

Determination of the initial and final moisture content of the grains

The moisture content was determined by the method of the oven with natural air circulation (105 + 3 °C for 24 h), in percentage (%) and dry basis (BRASIL, 2009).

Technological properties

Fissures formation

In order to determine the percentage (%) of fissured grains, samples of three replicates ($n = 3$), with 100 manually peeled rice grains, were visually evaluated with the aid of a system consisting of polarized light sheets (Polaroid brand, HN32 model, Brazil) with 9-W cold light source (LED), and digital image with magnification. To determine the head rice yield (HRY), samples composed of three replicates ($n = 3$), with 100 g of rough rice grains, were peeled and polished for 60 s, removing 8% of bran, in a Zaccaria machine (PAZ-1-DTA model, Limeira, SP, Brazil).

Head rice yield

Broken grains were removed by a machine and manually completed, according to the methodology indicated by Vanier et al. (2015).

Whiteness

Rice whiteness was obtained using a Zaccaria whiteness meter (MBZ-1 model, Limeira, SP, Brazil), with samples composed of three replicates ($n = 3$) of processed rice, and the results were expressed as rice whiteness percentage (%), according to a reference standard and previous calibration.

Determination of glass transition curve

The glass transition curve of the rice grains, cultivar 'Guri Inta' (long-thin class), was determined based on the methodologies proposed by Perdon et al. (2000), in which samples composed of three replicates ($n = 3$) of whole flour derived from grains dried at low temperature (25 °C in the grain mass), manually peeled and ground in experimental mill, were evaluated in relation to the sudden changes of heat flux, using a Differential Scanning Calorimeter (Shimadzu brand, DSC-60 model), under nitrogen atmosphere. Significant changes in heat flux (mW) measurements demonstrated by this device represented the glass transition temperature (T_g), for a particular condition of moisture of the sample (%) of the rice cultivar studied. To perform this analysis, samples of whole flour, with 20 ± 0.05 mg of dry mass, were placed in aluminum crucibles (100 microliter and 30 bar), subsequently sealed, with addition of certain amounts of distilled water, previously calculated, in order to obtain samples with different levels of moisture. The sealed crucible, containing sample of whole flour with specific moisture content, after resting for 24 h for homogenization, was heated in a DSC from 25 to 105 °C, using an empty crucible as reference. The glass transition temperature of the samples, in relation to the different levels of moisture, was determined from the midpoint of the abrupt change of the heat flux, observed in the DSC, with a heating rate of 3 °C per minute (SANDOVAL et al., 2009). These values were then plotted with respect to the respective levels of moisture (% dry basis), making it possible to construct the glass transition curve of the cultivar evaluated. Thereafter, the values of temperature and moisture content of the grains, for each drying and according to the thickness of the layer, were compared with this glass transition curve.

Cooking properties

Cooking time was determined using the Ranghino test, as indicated by Mohapatra and Bal (2006), in which about 100 mL of distilled water were boiled ($98 \pm 1^\circ\text{C}$) in a cup (250 mL), and 10 g of the sample of processed rice grains were placed in the boiled water. Cooking time was evaluated after 10 minutes and every 1 min from this point on. Ten rice grains were removed and pressed between two plates of clean glass to assess their degree of doneness. The cooking time was recorded when at least 90% of the grains no longer showed opaque or raw core. Then, 2 min were added to the final time recorded, for standardization. To determine the volumetric and gravimetric yields, the volume of rice grain samples (35 g) was measured. Subsequently, the sample was cooked in aluminum pans with 120 mL of boiled water ($98 \pm 1^\circ\text{C}$) according to the previously measured cooking time. After cooking, the samples were maintained for 30

min at $25 \pm 1^\circ\text{C}$, to determine the mass and the final volume (ARNS et al., 2014). The volumetric yield was determined using the following equation: volumetric yield (%) = (volume of uncooked rice divided by volume of boiled rice) multiplied by 100. Gravimetric yield was determined through the following equation: gravimetric yield (%) = (mass of uncooked rice divided by mass cooked rice) multiplied by 100.

Statistical analysis

The results were evaluated by analysis of variance (ANOVA), with three replicates and compared by Tukey test. Statistical significance was defined as $p < 0.05$.

RESULTS AND DISCUSSION

Drying curves as a function of layer thickness and temperature

The Figure 1 shows the influence of temperature and of layer on the drying curve

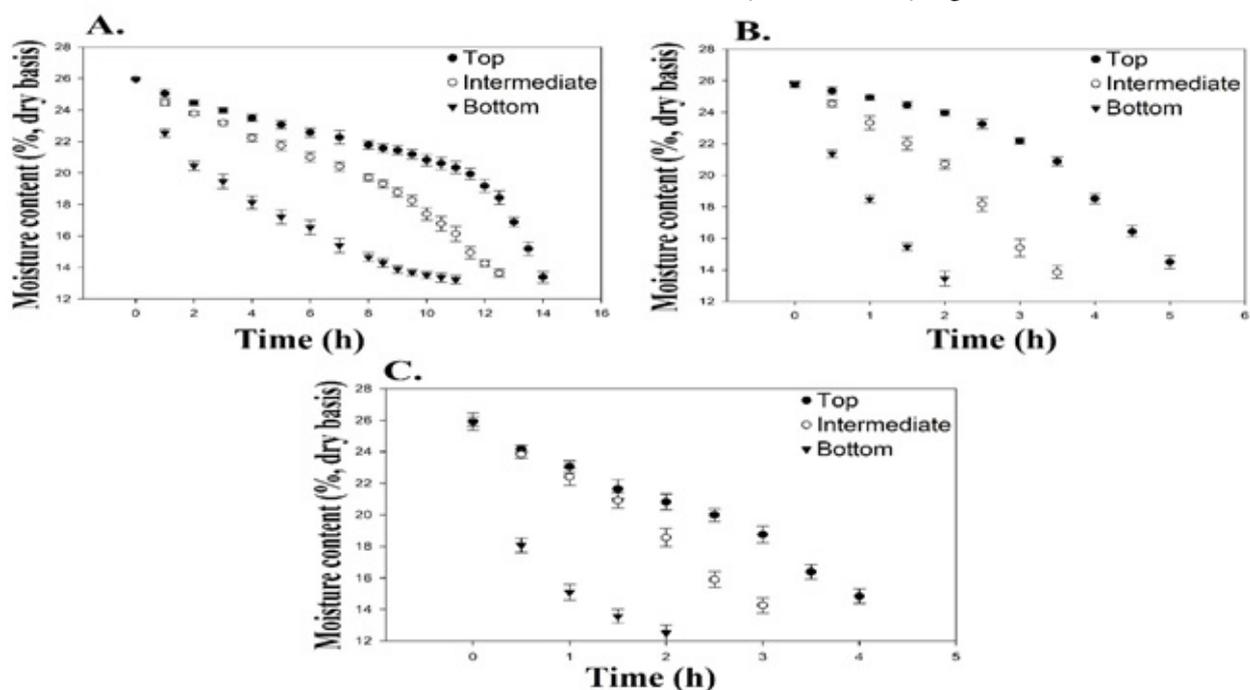


Figure 1. Drying curves of grains of rough rice (long-thin class) as a function of grain layer thickness (bottom of 0.00 to 0.15 m, intermediate of 0.15 to 0.30 m and top of 0.30 to 0.45 m) to drying with air temperature of: A.: 30°C , B.: 55°C , and C.: 80°C .

By comparing the drying curves (Figure 1), it is possible to observe that, for drying with average air temperature of 30°C , 14 h were necessary for the upper layer to reach the desired moisture content of 14% (dry basis). However, grains dried at an average air temperature of 55°C required a drying time of 5 h, and those dried at an average air temperature of 80°C

required only 4 h to complete the drying of this same layer. Therefore, drying with average air temperature of 80°C resulted in a 20% shorter drying time compared to grains dried at an average air temperature of 55°C , and 71% shorter drying time compared to grains dried at an average air temperature of 30°C . As demonstrated in other studies (SANDOVAL et al., 2009; DONG

et al., 2010), the increment of drying temperature intensifies the thermal changes, increasing the speed of moisture removal and reducing the drying time. The influence of rice layer thickness on the drying time was prominent, with difference of up to 3 h more for the top layer in comparison to the bottom layer. Non-uniformity of moisture distribution interferes with the drying rate, affecting the physical and structural characteristics of rice grains. In general, the lower layer showed a typical curve of thin-layer drying of grains, with a falling rate of drying. However, the upper layer had two phases. In the first phase, the moisture content decreased at a slower rate. As the drying front advanced, the moisture curve showed a deflection and subsequent increase in velocity of reduction of humidity until the end of the operation. The effects of layer thickness on rice drying was investigated by

Torki-Harchegani et al. (2014), who studied four layer thicknesses (each with 0.05 m), totaling a layer height of 0.20 m. The results indicated that the upper layers had higher moisture content. At the end of the drying process (time of 100 min, air temperature of 60 °C and an air speed of 0.9 m s⁻¹), there was a 4% difference in the moisture contents between the bottom layer (14%) and the top layer (18%). This kinetic behavior was similar to that found in this study, especially when drying occurred with temperature of 55 °C.

Glass transition curve applied to rice drying

The Figure 2 shows the values of temperature and moisture of the grains, for each drying condition and as a function of layer thickness (bottom, intermediate and top), compared to the glass transition curve of rice grains (Guri INTA).

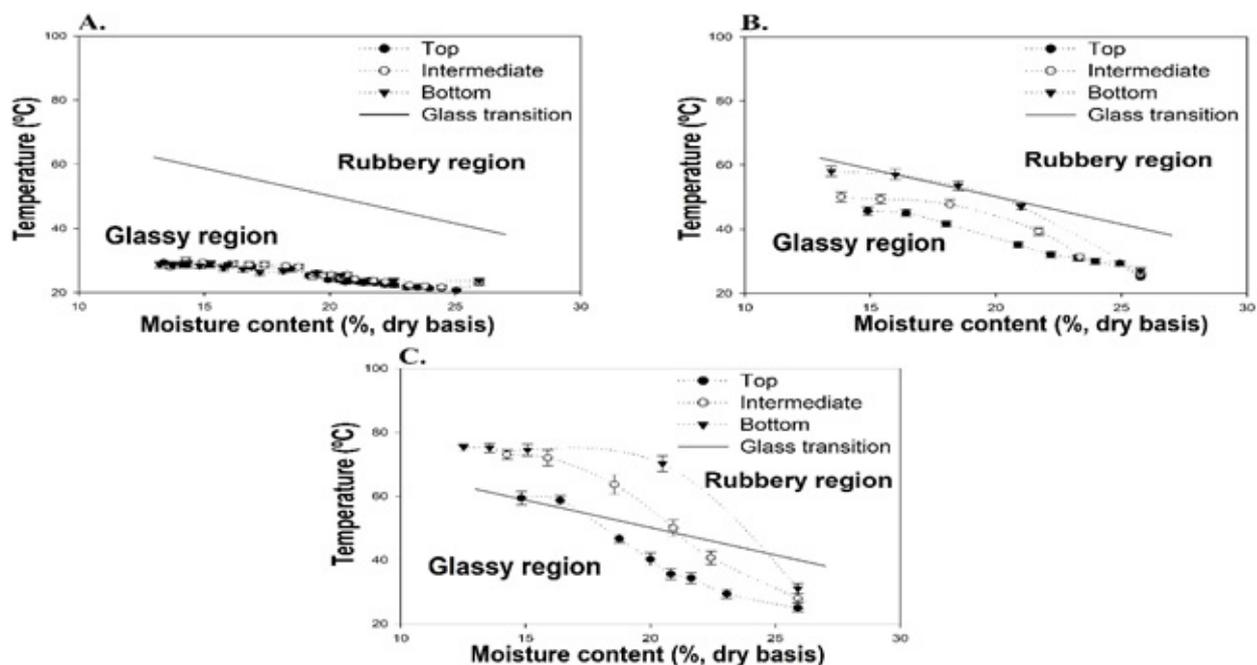


Figure 2. Glass transition curve and the values of temperature and moisture of grains of rough rice (long-thin class) as a function of grain layer thickness (bottom of 0.00 to 0.15 m, intermediate of 0.15 to 0.30 m and top of 0.30 to 0.45 m) to drying with air temperature of: A.: 30 °C, B.: 55 °C, and C.: 80 °C.

Figure 2 shows that the glass transition temperature (T_g) of the rice grains increased as the moisture content (MC) decreased. The polynomial equation of glass transition temperature [T_g (°C) = - 1.72 MC (%) + 84.52] was related to the moisture content, with high coefficient of determination ($R^2 = 0.98$). Chung et al. (2004) and Sandoval et al. (2009) emphasize that, in grains with high moisture content, the plasticizer effect exerted by water on rice starch is responsible

for reducing the value of glass transition temperature. In dry grains, a greater amount of heat and higher temperature are required for glass transition to occur. The glass transition curve of the studied cultivar (Guri Inta) shows that the glass transition temperatures were 50 °C for the sample with MC of 20% and 64 °C for the sample with MC of 12%. Schluterman and Siebenmorgen (2007) studied the glass transition temperature of rough rice, Francis and Wells cultivars

(long grains), using a glass transition curve in which, for MC of 10%, the glass transition temperature was 70 °C. Sandoval et al. (2009), using a DSC for determining the glass transition temperature of whole rice flour samples, with MC of 10.2%, found glass transition temperature of 65 °C. Heat and mass transfer in grain drying is a complex phenomenon, especially in drying procedures with large layer thickness. The water removal potential of the drying air is considerably reduced as it crosses the grain layer, promoting temperature and vapor pressure gradients that are directly proportional to the grain layer thickness, as shown in Figure 2. Additionally, in the drying with air temperature of 30 °C, the glass transition temperature had not been exceeded in any part of the grain layer, so the grains remained in the glassy state region (crystalline). In the drying with average air temperature of 55 °C, in the bottom layer, i.e. the closest one to the drying air entry, with moisture

between 20 and 15%, the temperature of the grains was equal to or a slightly above the glass transition temperature, indicating that there were changes in the physical state of some rice grains. On the other hand, in the drying with air temperature of 80 °C, in more than half of the grain layer, virtually along the entire drying operation, the temperature of the rice grains exceeded the glass transition temperature, indicating that a large part was subjected to change of physical state. These changes, when sufficiently large, can cause high tension gradients within the grain and, depending on the intrinsic characteristics of the cultivar, result in severe damage to its physical integrity, generating fissured grains and broken grains.

Fissures, head rice yield and rice whiteness

The influence of the tempering of 0, 120 and 240 min and drying with temperature of 30, 55 and 80 °C on some technological properties of rice can be observed in Table 1.

Table 1. Technological properties of grains of rice (long-thin class) as a function of temperature of the air of drying and tempering time.

Air temperature (°C)	Tempering (min)	Fissured kernels (%)	Head Rice Yield HRY (%)	Whiteness (%)
30 (control)	-	09.00 ± 2.58 d	64.44 ± 1.49 a	48.27 ± 0.50 a
55	0	19.00 ± 2.58 ab	40.55 ± 1.52 c	48.00 ± 0.35 a
55	120	16.00 ± 2.83 bc	45.77 ± 0.71 b	48.00 ± 0.10 a
55	240	12.00 ± 3.65 cd	63.26 ± 0.81 a	48.37 ± 0.25 a
80	0	23.00 ± 2.58 a	21.76 ± 0.16 e	48.10 ± 0.50 a
80	120	18.00 ± 2.83 abc	36.86 ± 1.18 d	48.07 ± 0.15 a
80	240	14.50 ± 1.91 bcd	43.07 ± 1.03 bc	48.03 ± 0.23 a

Mean ± standard deviation (n = 3). Lowercase letters in the same column indicate comparisons that did not have any significant differences as evaluated by Tukey's test at 5% probability ($p \leq 0.05$)

The results indicated that the drying with average air temperature of 30 °C led to the lowest percentage of fissured grains, whereas the drying with air temperature of 80 °C (55 °C in the grain mass), without tempering, resulted in the highest values. In the drying with air temperature of 55 °C (40 °C in the grain mass), the implementation of the tempering step, with a minimum of 240 min, resulted in 37% reduction in the percentage of fissured grains and a consequent increase of 56% in head rice yield (HRV), compared to the same drying temperature, but without tempering. This indicated that the tempering step promoted thermal and moisture equilibrium, and a reduction in

temperature, moisture and tension gradients generated during drying with high temperatures, leading to the reduction in the cracks that could occur in the grains. Dong et al. (2010) studied the tempering after the drying of rough rice (short and long grains) and concluded that the tempering of rice grains for 60 min reduced the incidence of cracks by approximately 50%. The drying at 30 °C resulted in the highest HRV (64.44 ± 1.49), and the drying at 80 °C, without tempering, resulted in the lowest yield (21.76 ± 0.16). The drying with average air temperature of 55 °C and tempering of 240 min led to head rice yield (HRV) equivalent ($p < 0.05$) to that of the drying at

average temperature of 30° C. Thus, it was observed that the higher the drying temperature employed, without proper tempering treatment, the greater the incidence of fissured grains and broken grains, and consequently, the lower the head rice yield (HRY). Rough rice grains subjected to drying temperatures above the glass transition temperature, without proper tempering treatment, do not resist high gradients of temperature and vapor pressure, which damage their physical integrity (TONG et al., 2019). Dong et al. (2010) observed that after drying of rough rice (short grains), the tempering for 120 min eliminated about 80% of the moisture gradient created, which was an important factor in the formation of cracks and subsequent broken of grains. About the whiteness, the results showed that drying and tempering steps did not significantly affect rice grains ($p < 0.05$). Madamba

and Yabes (2005) found similar results for the degree of whiteness.

Cooking characteristics

Table 2 shows the cooking properties as a function of drying temperature and tempering time. In general, the cooking time decreases with increasing drying temperature (from 14.32 to 12.11 min). Rice grains dried at air temperature of 55 °C, followed by the tempering step for 240 min, showed increased cooking time. The use of high drying temperatures (80 °C) increased the number of fissures in rice kernels (Table 1), which facilitated the absorption of water and reduced the cooking time. Mohapatra and Bal (2006) observed cooking time ranged between 12 and 24 min as a function of the polishing percentage, for three different rice cultivars.

Table 2. Cooking properties of grains of rice (long-thin class) as a function of temperature of the air of drying and tempering time.

Air Temperature (°C)	Tempering (min)	Cooking time (min)	Volumetric yield (%)	Gravimetric yield (%)
30 (control)	-	14.32 ± 0.36 a	330.12 ± 5.61 a	287.14 ± 12.37 a
55	0	12.15 ± 0.09 c	327.00 ± 11.47 a	279.52 ± 12.15 a
55	240	13.22 ± 0.22 b	319.05 ± 9.62 ab	283.33 ± 9.07 a
80	0	12.11 ± 0.01 c	306.02 ± 2.07 bc	271.90 ± 7.05 a
80	240	12.21 ± 0.09 c	296.82 ± 5.07 c	280.00 ± 6.54 a

Mean ± standard deviation (n = 3). Lowercase letters in the same column indicate comparisons that did not have any significant differences as evaluated by Tukey's test at 5% probability ($p \leq 0.05$).

There was a reduction of volumetric yield (Table 2) in the drying at air temperature of 80 °C, compared to the other treatments, and the volume of the grains after cooking was about three times larger than the volume of uncooked rice.

CONCLUSIONS

It was concluded through this study that: 1) grain layer thickness interfered with drying time, with a difference of up to 3 h more for the upper layer in comparison to the lower one; 2) implementing the tempering step has preserved the quality of rice grains, as the drying with temperature of air of 55 °C (40 °C in the grains) and tempering for 240 min led to results equivalent to those obtained in the drying of 30 °C (control).

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