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SINGLE-PASS DRYING OF ROUGH RICE USING GLASS TRANSITION PRINCIPLES

SINGLE-PASS DRYING OF ROUGH RICE USING GLASS TRANSITION PRINCIPLES

A dissertation submitted in partial fulfillment of the requirement for the degree of Doctor of Philosophy in Food Science

By

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> August 2012 University of Arkansas

ABSTRACT

The objective of this research was to study the drying characteristics, milling quality, and functional properties of rough rice subjected to single-pass drying while controlling kernel material states. Drying experiments were conducted at 60, 70, and 80°C and relative humidities ranging from 13 to 83%. High drying air relative humidities (> 63%) maintained both the kernel surface and core in a rubbery state during drying, whereas low drying air relative humidities caused rapid transitioning of the surface layers from a rubbery to a glassy state. Long-grain pureline cultivar, Wells, medium-grain pureline cultivar, Jupiter, and long-grain hybrid cultivar, CL XL729, were dried from harvest moisture content to 12.5% moisture content in a single-pass. Immediately after drying, samples were tempered in the drying container or in sealed plastic bags at the drying air temperature for 0, 30, and 60 min, after which they were spread in thin layers and cooled to ambient conditions. For all drying air temperatures and tempering conditions, milling quality was not significantly different from the controls when the relative humidity of the drying air was maintained above 63% (both the kernel core and surface maintained in a rubbery state during drying) and rice was tempered immediately after drying in sealed plastic bags and at the drying air temperature for at least 60 min. Minimal reduction to milling quality was observed at the low drying-air relative humidities when samples were tempered immediately after drying in sealed plastic bags and at the drying air temperature for at least 60 min: tempering in containers having large headspaces or for shorter durations failed to reduce the intra-kernel stresses created during drying due to differences in material state between the surface and the core, thereby causing kernel fissuring and breakage during subsequent milling. The high-temperature conditions did not affect color, degree of milling, and thermal properties. However, pasting viscosity profile was significantly affected.

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DEDICATION

This dissertation is dedicated to the Rice Processing Industry and the Grain Industry at large.

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Ondier, G. O., Siebenmorgen, T. J., and Mauromoustakos, A. 2012. Physicochemical properties of rough rice dried in a single pass using high temperatures. Cereal Chemistry, *To be submitted*.

I. INTRODUCTION

Rice is among the oldest of cultivated crops and currently ranks as one of the most important foods in the world. Rice constitutes the staple food for a large proportion of the world's population, and provides income to millions of farmers throughout the world (Evanson et al., 1993). Rice is mostly consumed as whole kernels and its consumers represent one of the most demanding cereal markets with regards to product quality. Kernel quality is of paramount importance to the rice processing industry because most of the rice produced is destined for human consumption.

Two of the main physical indices used to determine rice quality are head rice yield (HRY) and degree of milling. Head rice yield is accepted as the current measure of commercial physical quality and is defined as the mass percentage of rough rice that remains as head rice (kernels that are at least 3/4 of the original kernel length) after complete milling. Broken kernels are more susceptible to insect and microbial attacks, decreased seed germination rates, and the market price for broken kernels may be much less than that for whole kernels (Li et al., 1999). Other rice quality parameters frequently reported include pasting properties, chemical properties, and sensory quality (Daniels et al., 1998; Meullenet et al., 1999; Pearce et al., 2001; Perdon et al., 1997; Ranalli et al., 2003, and Zhou et al., 2003).

Post-harvest management of rice has been reported to play an essential role in maintaining HRY and other quality parameters. In various production areas, there are still significant post-harvest losses due to inadequate post-harvest facilities and methods, especially drying. Rough rice is normally harvested from the field at 14–24% moisture content (MC¹) to prevent field loss due to lodging and shattering, and reduce handling costs (De Padua et al.,

¹ Unless otherwise specified, all moisture contents are reported on a wet-basis.

1985). High post-harvest temperatures and relative humidities (RHs), coupled with high MC of rough rice, encourage mold growth and a high respiration rate in grain. High respiration rates reduce the dry matter of rice and may produce sufficient energy to be detrimental to product quality, while molds may produce deadly toxins (Soponronnarit and Chinsakolthanakorn, 1990). Therefore, the MC of rough rice must be reduced to less than 13% for safe long-term storage.

Open-air sun drying, one of the oldest methods of drying grains, is still operational in developing countries (Amir, 1972). Solar energy is preferred to other sources of energy because it is abundant, inexhaustible and non-polluting. Solar energy can be harnessed at relatively low cost and has no associated environmental dangers. Large-scale production however limits the use of open-air sun drying due to the inability to properly control the drying process, weather uncertainties, high labor cost, large area requirements, mixing with dust and other foreign material, and insect infestation (Basunia et al., 2000). Solar drying systems must be properly designed in order to meet particular drying requirements of specific crops (Exell, 1980). As such, numerous attempts have been made in recent years to better harness solar energy for use in drying grain. Many researchers have proposed the use of simple dryers in which heated air rises by natural convection through the grain to dry rough rice in small batches of 0.5 to 1 ton (Wieneke, 1977; Exell and Kornsakoo, 1978; Exell, 1980; Phongsupasamit, 1981; Boonthunjinda, 1980).

Another form of drying, which is mostly practiced on-farm is natural air drying. Natural air is used in grain drying systems where air is blown by a fan through grain stored in a bin. The use of natural air in drying grains stored in a bin reduces the labor cost and helps in maintaining grain quality (Huffman, 1980). However, the method is not entirely reliable because it depends on the ambient conditions, which are unpredictable. The difficulties in natural air drying of

grains are exacerbated during the night because of sharp drops in temperature that result in high RHs (> 85%) (Bradburn et al., 1993; Miller, 1985). High RH conditions may cause wetting of dried grains, subsequently leading to increased respiration, mould growth and mycotoxin formation, germ damage, mustiness and caking (Odigboh, 1976; Smith et al., 1985; Thoruwa and Abdallah, 1988). To lower the RH and improve the moisture holding capacity of the drying air, a gas or propane burner is sometimes used to raise the incoming air temperature by 5-10°C (Ryniecki et al., 1991). Heating is only required when the RH of the incoming air is above 60% (Huffman, 1980). Heating of incoming air when the RH is less than 60% will over-dry the grain and adds unnecessary cost to the drying process. To ensure high HRYs and germination rates, the drying air temperature is recommended not to exceed 43°C (Soponronnarit & Preechakul, 1990). Increasing the airflow rate helps in reducing hot spots and increases the rate of moisture removal from a bulk of grain (Huffman, 1980). Though sun-drying and natural-air drying are inexpensive and result in less reduction in milling quality, the drying durations are long and impractical for facilities handling large volumes of high moisture rough rice.

In most commercial facilities, drying operations utilize forced convection of heated air to dry grain. Heated air grain drying, as widely used in the commercial rice industry, employs high temperatures in different drying methods and dryer designs (Inprasit and Noomhorm, 2001). Column and mixed flow dryers generally operate at 45 - 78°C (Calderwood et al., 1975; Hogan et al., 1958). Some multi-stage dryers have been reported to operate at temperatures as high as 80 - 200°C (Inprasit and Noomhorm, 2001). In high temperature drying, the MC of rough rice is reduced from more than 20 - 18% in the first stage of drying, followed by a second stage that reduces the MC to 12 - 14% (Ali et al., 1980; Jindal et al, 1986).

Fast rate drying is more efficient and costs less per unit of moisture removed. However, rapidly drying rough rice using high temperatures may lead to kernel fissuring and eventual breaking during milling (Inprasit and Noomhorm, 2001). According to Bonazzi et al. (1997), rough rice quality can be seriously damaged if air with high evaporative capacity is used for drying, this being a function of temperature and RH. Kunze et al. (1985) suggested that it was the drying rate, more so than the drying air temperature, that determined the final rice quality. Studies have shown that a single percentage point change in MC produces stresses in the grain that are 100 times the magnitude produced by a 1°C temperature change.

Given the influence that high-temperature drying has on milling quality, end-use product functionality, and commercial value of the rice crop, it is important to optimize the application of high temperature conditions by developing drying methods that are equally effective in drying rough rice as well as maintaining final product quality. The primary focus of this dissertation was developing a new method of drying rough rice at high temperatures that would prevent kernel fissuring and minimize the deleterious effects of the drying condition on end-use product functionality. A second focus was developing proper tempering methods, which is a key step in high temperature drying that could have significant implications on final product quality. Finally, the effect of the high-temperature drying and tempering conditions on end use product quality was determined. The specific objectives of this dissertation were as follows:

1. Equilibrium moisture content studies

The experiments were designed to describe the high-temperature drying characteristics of rough rice. The objectives were to, 1) measure the desorption isotherms of long-grain rough rice subjected to elevated drying air temperatures in a laboratory-scale, fluidized-bed system; 2) use the Page equation to describe the drying data thus estimating the drying constants k and n. and 3)

evaluate the appropriateness of the Modified Chung-Pfost equation for estimating equilibrium data of rough rice for the range of temperatures and RH studied.

2. Milling quality studies

These experiments investigated the potential of fast rate drying at high temperatures while maintaining milling quality. The main objective was to develop a method that could be used to dry rough rice in a single pass with minimal reduction to milling quality. This approach could make the commercial drying process more efficient by reducing the number of passes and tempering steps.

3. End-use product functionality studies

The experiments were designed to evaluate the effect of single-pass drying at high temperatures on end-use product functionality. Physicochemical properties, namely, color, degree of milling, pasting viscosity profiles, and thermal properties were evaluated.

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II. LITERATURE REVIEW

Rice drying

High drying air temperatures are used in commercial, cross-flow systems to dry rough rice (Kunze, 1979; Schluterman and Siebenmorgen, 2004). Heated air facilitates shorter drying durations, but has the potential of reducing milling quality if not properly implemented (Kunze, 1979; Schluterman and Siebenmorgen, 2004). Various steps must, therefore, be undertaken to preserve kernel quality when drying rough rice at high temperatures. Soponronnarit et al. (1990) recommended that the drying air temperature should not exceed 60°C. Abud-Archila et al. (2000) showed that high drying temperatures, up to 60°C, can be used without reducing the processing quality of rice when the drying air RH is high. For commercial application, it is important to understand high temperature drying processes and how they affect the rice kernel with regards to fissure formation, thus controlling and optimizing the drying conditions.

Rice kernel fissuring due to high temperature drying

Several studies have shown that kernel fissuring may result from severe drying conditions or exposure of dried rice kernels to air at high RH (Kunze and Prasad, 1978, Kunze 1979, Sharma and Kunze, 1982, Fan and Marks, 1999). Kernel fissuring is a major concern in rice drying because fissured kernels affect the functional properties of rice and are more susceptible to breakage during the milling process (Cnossen et al., 2001).

Studies have shown that high temperature drying establishes a MC gradient between the surface and the center of the kernel, i.e. as moisture evaporates from the outer layers of the kernel, moisture gradients are established within the kernel (Siebenmorgen et al., 2004). Moisture gradients developed during drying result in tensile and compressive stresses within the kernel, which if sufficiently large, provoke kernel fissuring and breakage (Ban, 1971; Kunze and

Choudhury, 1972; Sharma and Kunze, 1982; Nguyen and Kunze, 1984; Abud-Archila et al., 2000, and Cnossen et al., 2003). The glass transition principle may help explain the effect of moisture gradient on fissuring trends seen after high temperature drying (Cnossen et al., 2001). As a kernel of rice is subjected to high temperatures, starch transitions from a 'glassy' to a 'rubbery' state. This transformation from a glassy to rubbery material is referred to as glass transition and the temperatures within which the transition occurs are known as glass transition temperatures (T_g). The T_g of brown rice kernels has been determined to follow an inverse relationship with MC (Perdon et al., 2000; Sun et al., 2002; Siebenmorgen et al., 2004) and ranges from 24°C to 78°C (Fig. 1).



Figure 1: State diagram for Drew and Bengal brown rice kernels with the fitted glass transition line determined by Siebenmorgen et al. 2004.

Perdon et al. (2000) showed that the physical properties of a rice kernel changed dramatically as the kernel temperature increased above T_g such that, at a temperature and MC below T_g , starch existed as a 'glassy' material, with low expansion coefficients, specific volume, and diffusivity while above T_g , starch existed as a rubbery material with much higher expansion coefficients, specific volume, and diffusivity.

Cnossen and Siebenmorgen (2000) hypothesized that fissuring of rice mainly occurred from the surface, midpoint, and center of the kernel as it crosses the T_g line upon cooling. They proposed that as major portions of the kernel remained at a high MC and in a rubbery state during drying, the surface could be transitioning into the glassy state due to a much lower MC (Figs. 2 and 3). Upon cooling, greater portion of the kernel may cross the T_g line, transitioning into the glassy state. The difference in material states of the center and surface of the kernel during drying and cooling determines the extent of fissuring due to the different mechanical properties exhibited by the kernel in the glassy state and rubbery state (Perdon et al., 2000).



Figure 2: Hypothetical temperature (T) and moisture content (MC) gradient in a rice kernel plotted onto a rice kernel (adopted from Cnossen et al. 2000).



Figure 3: Hypothetical moisture content gradient in a rice kernel during drying plotted onto a state diagram (adopted from Cnossen et al. 2000 and updated with the glass transition line from Siebenmorgen et al. 2004).

Tempering procedures, whereby rice is held in bins at constant temperature for a given duration between drying passes to allow MC equilibration within kernels, are employed in commercial drying to eliminate moisture gradients (Fig. 4). Multiple drying passes, which reduce the MC by small amounts (two to three percentage points) with each drying pass, are employed between tempering periods (Kunze and Calderwood, 1985). Cnossen et al. (2000) has shown that high temperatures, up to 60°C, can be used to dry rough rice as long as sufficient tempering is allowed. Wiset et al. (2005) suggested that the enhancement of kernel toughness during high temperature drying and tempering may be due to partial gelatinization inside the kernel when the MC and temperature is appropriate.



Figure 4: Alternative responses of the various sections of a rice kernel during tempering (adopted from Cnossen et al. 2000 and updated with the glass transition line from Siebenmorgen et al. 2004).

Though tempering may help eliminate moisture gradients developed within the kernels, thus prevent fissuring, the high temperatures used (> 40° C) in conventional cross-flow drying systems still result in milling quality reduction. In addition to milling quality, high-temperature drying can affect other quality parameters such as color, pasting viscosity profile, cooking properties, aromatic properties etc.

Effect of high temperature drying on rice sensory quality

Because rice is mostly consumed as whole brown or white kernels, the sensory attributes associated with different rice cultivars are very important to consumers. Variations in the sensory properties can make the product desirable or unacceptable. Drying has been identified as one of the major factors influencing sensory attributes in processed rice products.

Color

Rice whiteness has been found to decrease with increasing grain drying temperatures and drying durations (Bunyawanichakul et al., 2005). Dillahunty et al. (2001) showed that yellowing of rice increases with increasing exposure to high temperatures (> 45°C). Chemical and physical transformations induced by heating (Maillard reaction), and translocation of color from the rice husk and bran to the endosperm may cause discoloration (Inprasit and Noomhorm, 2001). Longer drying durations, and high initial MCs during drying, accelerate the Maillard reaction that may lead to discoloration (Inprasit and Noomhorm, 2001). Sun-drying and the drying methods using air at 30 and 40°C have been found to result in a greater degree of whiteness of the milled rice compared to samples dried at 70°C (Sugunya et al., 2003). Soponronnarit and Chinsakolthanakorn (1990) proposed that high post-harvest temperatures may lead to increased enzyme activity, which accelerate rice yellowing.

Pasting viscosity profile

Though reports are conflicting, many studies have shown that temperatures above a certain level influence the peak viscosity of rice (Ban, 1971; Dillahunty et al., 2001; Wiset et al., 2001; Meeso et al., 2004). Ban (1971) determined that the reduction in peak viscosity of high temperature dried rice samples was similar to that of parboiled rice. Dillahunty et al. (2001) observed significant reduction in peak viscosity for rice samples exposed to 66, 70 and 92°C for durations greater than 12 h. Wiset et al. (2001) observed significant decreases in peak viscosity of rice dried at 85 – 90°C compared to in-store dried rice. Meeso et al. (2004) showed a reduction in peak viscosity for rice dried at 150°C for 1.5 min. All pasting property changes were more pronounced at higher drying temperatures; for instance, setback and pasting temperatures had increasing trends while peak viscosities, trough, breakdown, and final viscosities decreased at higher drying temperatures (Wiset et al., 2007). The peak viscosity was found to be greater for low temperature dried rice samples than for high temperature dried samples (Daniels et al., 1998).

Cooking properties

The use of high drying temperatures ($85 - 150^{\circ}$ C) has been reported to result in increased water absorption (with tempering between drying passes) and decreased water absorption (without tempering) compared to in-store drying methods (Wiset et al., 2001). Imprasit and Noomhorm (2001) reported a decrease in water absorption when rice was dried at $120 - 150^{\circ}$ C, with or without tempering. Rice dried at low temperatures (< 33°C) has been reported to have greater water absorption and volume expansion than did rice dried at high temperatures (> 53°C) (Daniels et al., 1998). Hardness of cooked rice was reported to increase with increase in drying temperatures (Imprasit and Noomhorm, 2001). High drying temperatures and longer drying

durations may cause partial gelatinization of starch granules, resulting in accelerated ageing that affects the grain qualities in ways similar to parboiling of rice (Jindal et al., 1986). Cooked rice kernels from samples previously dried at high temperatures are softer, more cohesive, exhibit higher starchy note, and have lesser overall impact than samples dried at low temperatures (Meullenet et al., 1999; Champagne et al., 1998).

Aromatic properties

A large number of volatile compounds that would contribute to the aroma of fresh rice have been observed in cooked and uncooked rice (Yajima et al., 1978). Buttery et al. (1982) identified, and Jezussek et al. (2002), Laksanalamai et al. (1993) and Paul et al. (1989) confirmed, 2-acetyl-1-pyrroline as the most important compound contributing to the aroma of rice. Sugunya et al. (2003) reported low concentration of 2-acetyl-1-pyrroline, and an increase in off-flavors (*n*-hexanal and 2-pentyl furan) in rice samples dried using high temperatures (> 70°C). Wiset et al. (2007) found low-temperature drying to better preserve the amount of 2acetyl-1-pyroline than high temperatures. Wangpornchai et al. (2004) postulated that volatile compounds evaporate from rice during high temperature drying.

Moisture desorption isotherm and prediction models

A thorough knowledge of moisture diffusion kinetics and the influence of temperature and RH are important when conducting drying studies. Newton's law of cooling can be used to describe the moisture loss in grain whereby the drying rate (dMC/dt) is proportional to the difference between the average MC in solid material and the EMC (Wongweis et al., 2000).

$$dMC / dt = -k (MC - EMC)$$
(1)

If the drying constant, k, is considered independent of the average MC and the EMC, the equation may be integrated to:

$$MR = \exp(-k t)$$
 (2)

where MR is (MC - EMC)/(IMC - EMC) and IMC is initial moisture content.

This model is also called the exponential model and has been utilized by many researchers to describe the drying of biological materials (White et al., 1981; Shei et al., 1998). Page (1949) developed a modification of the equation (2) to the form of:

$$MR = \exp(-kt^{n})$$
(3)

Where MR is moisture ratio and *t* is the drying duration (h).

Page's equation has been used to describe thin-layer drying characteristics for wheat (Becker and Sallans, 1957) and rough rice (Agrawal and Singh, 1977; Basunia et al., 1998). The constants in these equations have no physical meaning and are determined by fitting a curve to the experimental data.

Temperature and RH, and their influence on EMC

To determine the EMC at the process temperature and RH, desorption isotherms are required. A sound knowledge of the relationship between the EMC and the equilibrium RH (ERH), for a given temperature, is important to fully describe the drying process, besides the intrinsic drying kinetics, which are normally expressed in thin-layer drying equations (Sun and Woods, 1994).

The modified Henderson, modified Chung-Pfost, and modified Halsey equations are commonly used to fit the EMC/ERH data of grains and seeds (Chen, 1989). The modified Handerson equation (MHE) (Thompson, et al., 1968) and the modified Chung-Pfost equation (MCPE) (Pfost, et al., 1976), which are provided by the ASABE Standards (ASABE, 1995), are highly recommended for describing sorption behavior of agricultural material. Many researchers have used the MCPE and the MHE to describe rough rice data and found them to represent the

data well (Agrawal and Singh. 1984: Zuritz et al., 1979: Banszek and Siebenmorgan, 1990). Each of the two equations has three parameters and can be solved explicitly for RH as a function of temperature and MC, or for MC as a function of temperature and RH. The modified Chung-Pfost equation (MCPE) (Pfost, et al., 1976) can be written as:

$$R_{\rm H} = \exp\left[-\frac{C_1}{T+C_2} \exp\left(-C_3 M\right)\right],$$

$$M = C_1 - C_3 \ln\left[-\left(T+C_2\right) \ln\left(R_{\rm H}\right)\right].$$
(4)

The modified Henderson equation (MHE) (Thompson et al., 1968) can be written as

$$1 - R_{\rm H} = \exp\left[-C_1(T + C_2)M^{C_3}\right],$$

$$M = \left[\frac{\ln\left(1 - R_{\rm H}\right)}{-C_1(T + C_2)}\right]^{1/C_3}.$$
(5)

In equation 4 and 5, R_H is the equilibrium relative humidity in decimal, M is the equilibrium moisture content (decimal dry-basis), T is the temperature in ^oC and C₁, C₂, and C₃ are equation coefficients.

Of the two equations, the most commonly used is the MCPE and has been used extensively to describe rough rice data (Pfost, et al., 1976). The results from Pfost et al. are adopted as ASABE Standard, D245.4 (ASAE, 1995) and the form of the equation used is:

$$EMC = 29.394 - 4.6015 \ln \left[-(T + 35.703) \ln (RH) \right], \tag{6}$$

Where T is the temperature (°C) and RH is the relative humidity (decimal).

Energy balances during drying.

The heat and mass transfer processes occurring during rough rice drying are different from other cereal grains because rough rice has an outer husk cover and a bran layer. It is therefore important to determine the moisture diffusion kinetics in rough rice and how they are influenced by drying conditions. Saponronnarit (1998) derived the energy equation for a thin layer of rice based on the first law of thermodynamics by assuming that heat loss from the layer periphery was very small and can be considered negligible. Equating the sum of changes of the internal energy in the control volume of a thin bed and enthalpy of flowing stream to zero gives the following equation:

$$c_{a}T_{o} + (2502 + c_{v}T_{o})H_{o} + Rc_{pw}T_{grain} = c_{a}T_{f} + (2502 + c_{v}T_{f})H_{f} + Rc_{pw}T_{f}$$
(7)

where c is the specific heat capacity (kJ/kg °C), T is the air temperature (°C), T_{grain} is the grain temperature (°C), H is the absolute humidity (kg H₂O/kg dry air), and R is the ratio of dry grain mass to dry air mass (kg dry matter/kg dry air) with subscripts: '*a*' representing dry air, '*f*' after drying, '*o*' before drying, '*pw*' the wet grain, and '*v*' the water vapor.

Preechakul (1986) developed an equation quantifying the shrinkage of bulk long-grain rough rice with validation using rough rice with MCs from 12.0 to 39%, which can be written as:

$$v = 0.001997 + 0.0012 \text{ MC}$$
(8)

Where *v* is the volume per kilogram of dry matter (m^3/kg) , and MC is the moisture content of rough rice (decimal dry-basis).

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III. CHAPTER ONE

Equilibrium Moisture Contents of Rough Rice Dried Using High-Temperature, Fluidizedbed Conditions

ABSTRACT

Desorption isotherms of long-grain rough rice with initial moisture content of 20.6% (wet-basis) and dried in a fluidized-bed system at temperatures ranging from 60 to 90°C and relative humidities from 7 to 75% were measured. Rice sample mass and drying air conditions were recorded throughout the drying duration for each test until a steady-state mass was attained. The Page equation, with experimentally-determined drying parameters, was used to describe the drying data. Equilibrium moisture contents were used to estimate empirical constants of the Modified Chung-Pfost equation. The resulting Modified Chung-Pfost equation described experimental data with a root mean square error, RMSE, of 0.73 and a coefficient of determination, R^2 , of 0.94.

INTRODUCTION

The main consideration in commercial drying of rough rice is reducing moisture content (MC^2) in the shortest duration possible without adversely affecting milling quality. For the year 2008 – 2009, approximately 9.26 million tons of rice was harvested in the US (USDA, 2010). These high production rates were supported by greater combine and transport capacities, which in turn placed increased pressure on driers. Increased drying speed, afforded by shorter drying durations, would enable drying facilities to increase throughput and thus meet the need to dry increasing crop influx.

² Moisture content is expressed on a wet basis unless specified otherwise.

A recent approach to rapid drying of high-MC rough rice utilizes high-temperature, fluidized-bed drying conditions (Soponronnarit et al., 1996: Huang-Nguyen et al., 1999). This technology offers several features: 1) an even flow of fluidized kernels permits continuous, large-scale operations with ease of product handling, 2) high heat and mass transfer rates create rapid movement of moisture from individually exposed kernels to air, and 3) rapid mixing of fluidized kernels leads to uniform drying throughout the fluidized-bed, thus enabling better control of the drying process (Hovmand, 1987).

Fluidized-bed drying is being used commercially for drying rice in Asia. In fluidized beddrying, air temperature and bed thickness have been shown to be the main factors affecting rough rice drying rate (Tumambing and Driscol, 1991) while milling quality and color were dependent on the MC at which rough rice exited the drier (Sutherland and Ghaly, 1990; Wetchacama, et al., 2000). Drying air temperatures ranging from $140 - 150^{\circ}$ C, air velocity from 2.0 - 3.0 m/s, and bed thickness of up to 10 cm have been recommended for fluidized-bed drying of high-MC rough rice (higher than 23%) to maximize efficiency while maintaining quality (Soponronnarit et al., 1994). Fluidized-bed drying has, however, not been accepted in the United States. In order to facilitate this possible acceptance, research is needed to fully quantify the kinetics of fluidizedbed rice drying under varying temperature and relative humidity (RH) conditions using U.S. rice cultivars. A key property necessary for this quantification is equilibrium moisture content (EMC) corresponding to a given temperature and equilibrium relative humidity (ERH) (Sun and Woods, 1997a and 1997b).

Equilibrium moisture content, defined as the MC at which hygroscopic particles are neither gaining nor losing moisture, is dynamic and depends on the temperature and RH of surrounding air. The two basic techniques used to measure EMC of foods and agricultural

materials are the manometric and gravimetric methods (Gal 1975, 1981). Manometric methods are based on vapor pressure measurements in the sample environments while gravimetric methods measure changes in mass.

Many studies have employed the use of mathematical models to describe EMC/ERH/Temperature relationships in complex drying systems (Iguaz and Versada, 2007, Aviara et al., 2004; Basunia and Abe, 2001; Chen and Morey, 1989; Sun and Byrne, 1998; Sun and Woods, 1994). The American Society of Agricultural and Biological Engineers Standard D245.6 (ASABE, 2007) lists the Modified Chung-Pfost (Chung and Pfost, 1967), Modified Halsey (Chirife and Iglesias, 1978), Modified Henderson (Henderson, 1952), Modified Oswin (Chen, 1988, Oswin, 1946) and the Guggenheim-Anderson-deBoer (GAB) as equations that can be used for modeling grain sorption equilibria data.

Studies have reported the Modified Chung-Pfost and Modified GAB equations as the most satisfactory theoretical isotherm/EMC models (Iguaz and Versada, 2007, Multon, 1998, Van der Berg, 1984, Weisner, 1985, Speiss and Wolf, 1987). Iguaz and Versada (2007) reported the Modified Chung-Pfost and Modified GAB models to be most appropriate in estimating EMC of rough rice at temperatures ranging from 40 to 80°C, Basunia and Abe (2001) reported the Modified Chung-Pfost as the best in the range of 12 to 51°C, while Sun (1999) identified the Modified Chung-Pfost as most appropriate for describing EMC/ERH sorption isotherms for wheat. The Modified Chung-Pfost equation is given as follows;

$$M_{e} = \frac{-1}{B} Ln \left[\frac{-(T+C)LnRH}{A} \right]$$
(1)

Where RH is relative humidity in decimal form, T is temperature in $^{\circ}$ C, M_e is equilibrium moisture content in % dry-basis, and A, B, and C are grain-specific empirical constants. Moisture content dry-basis is given by;

$$MC_{dry-basis} = \frac{100 \times MC_{wet-basis}}{100 - MC_{wet-basis}}$$

Considering that fluidized bed drying utilizes drying air temperatures that are not commonly used in the U.S. drying industry, there is need to evaluate the Modified Chung-Pfost equation for estimating EMC at these high temperature conditions. Therefore, the objectives of this study were to, 1) measure the desorption isotherms of long-grain rough rice subjected to elevated drying air temperatures in a laboratory-scale, fluidized-bed system; and 2) evaluate the appropriateness of the Modified Chung-Pfost equation for estimating equilibrium data of rough rice for the range of temperatures and RH studied.

MATERIALS AND METHODS

Test System

A 0.91-m³ (32-ft³) environmental chamber (Platinous Sterling Series T and RH Chamber, ESPEC North America, Hudsonville, MI) was utilized to produce drying air at set temperature and RH conditions (Fig. 1). The chamber was capable of maintaining air conditions at set levels within a range of temperatures (-35°C to 150°C) and RHs (6% to 98%). The air in the oven was circulated at 0.38 m³s⁻¹ and conditions were monitored using a digital temperature and RH probe (Hygro-M2, General Eastern, Woburn, MA). The digital RH probe was calibrated using Potassium Sulfate, Sodium Chloride, and Lithium Chloride (Merck, Darmstadt, Germany) prior to the start of the experiment.

A metal cylinder, 20.3 cm (8 in) in diameter and 61.0 cm (24 in) tall, with a perforated floor to hold rice samples for drying, was mounted to a metal plenum; this drying apparatus was placed inside the environmental chamber. The drying cylinder was wrapped with 2-mm (0.02-in)

thick, ceramic fiber insulation (Zirconia Felt ZY-50, Zircar Zirconia Inc., Florida, NY). A 25.4cm (10-in) diameter centrifugal fan (4C108, Dayton Electric Manufacturer Co., Chicago, IL), coupled to a 0.56-kW (0.75-hp), three-phase electric motor (3N443BA, Dayton Electric Manufacturer Co., Niles, IL), was mounted outside the chamber to avoid high temperature exposure. This fan suctioned air at set temperature and RH from the chamber through a port located in the chamber wall, and then passed the exhaust air through a second port in the chamber wall connected to the plenum beneath the drying cylinder. The desired airflow rate through the drying cylinder was achieved by regulating the electrical frequency of the fan motor using a frequency inverter (AF-300 Mini, GE Fuji Drives USA, Salem, VA), which controlled the motor and fan shaft rotational speed.

A spring-loaded damper constructed in the plenum controlled airflow direction by either diverting air through the perforated floor, or closing off the perforated floor, allowing the air to empty into the environmental chamber. Opening and closing of the spring-loaded damper was controlled by a linear actuator (damper actuator) (LACT4P, SPAL USA, Ankeney, IA) mounted outside the environmental chamber and connected to the damper by a cable that passed through a port in the chamber ceiling. A second linear actuator (load cell actuator) (LACT4P, SPAL USA, Ankeney, IA) mounted outside the environmental chamber, and directly above the drying cylinder was coupled to a 178-N (40-lb_f) full-bridge, thin-beam load cell (LCL-040, Omega Engineering Inc., Stanford, CT) that was attached to the drying cylinder via a cable that passed through a second port in the chamber ceiling.

At specified durations, the damper actuator was activated to raise the spring-loaded damper, thereby preventing airflow through the rice sample. The load cell actuator was then activated to suspend the drying cylinder just above the drying apparatus plenum. After a

stabilization period, the mass of the drying cylinder and sample was recorded as a milli-volt signal from the load cell. Voltage data were converted to masses using a previously-established calibration. The weighing procedure, which lasted 30 s, was repeated at selected intervals during a drying trial until masses remained approximately constant, varying by less than 0.01 g. Temperatures and load cell data were collected using a data logger (21X Micrologger, Campbell Scientific, Salt Lake City, UT) and stored onto a computer (Latitude C810, Dell Computer Corp., Round Rock, TX).



Figure 1: Schematics of the high-temperature, fluidized-bed drying system.

Measurement of Airflow Rates

Prior to drying trials, fan performance tests were conducted using a metal duct, 15.2 cm (6 in) in diameter and 1.8 m (6 ft) in length, to establish the fan motor electrical frequency-airflow rate relationship for the circulation fan. Airflow rates were adjusted by varying fan motor electrical frequencies from 40 to 60 Hz. Average air velocity in the test duct was measured using a hot wire anemometer (8450-13E-V-STD-NC, Control Company, Friendswood, TX).

Also prior to drying trials, the airflow rate/electrical frequency needed for fluidization of a 5.1-cm (2-in) rough rice bed in the drying cylinder was determined. The procedure of Subramani et al (2007) was used to determine minimum fluidization velocity at 60, 70, 80, and 90°C. A known mass of rice required to attain a 5.1-cm (2-in) grain depth, was poured into the drying cylinder. The fan was turned on and air velocity increased to fluidize the bed of rice vigorously. The air velocity was then slowly decreased until the fluidized bed was reduced to a fixed bed. The pressure drop at different depths across the 2-in rice bed was determined by inserting a static pressure probe from the top of the cylinder. At minimum fluidization velocity, the pressure drop across the bed was constant and the weight of rice was supported by the air flow (Subramani et al. 2007).

Rice Samples

Long-grain rice (Cybonnett) was harvested at the University of Arkansas Northeast Research and Extension Center near Keiser, AR on 8/28/07 at approximately 20.6% MC. The rice was cleaned using a dockage tester (XT4, Carter Day Co., Minneapolis, MN) and placed in storage at 4°C for two months. Prior to each drying trial, samples were withdrawn from storage, sealed in plastic bags, and allowed to equilibrate to room temperature (20°C) overnight. The MCs of the rice samples were then measured by drying duplicate, 15-g (0.033-lb_m) samples for

24 h in a convection oven (1370 FM, Sheldon Inc., Cornelius, Oregon) maintained at 130°C (Jindal and Siebenmorgen, 1987).

Drying Procedure

A 1.11-kg (2.45-lb_m) rice sample, which was required to attain a 5.1-cm (2-in) grain depth, was placed in the drying cylinder. A screen was placed on top of the cylinder to prevent inadvertent removal of fluidized kernels. The drying apparatus was then placed inside the environmental chamber and attached to the load cell actuator. The chamber control system was then activated to establish desired temperature and RH air conditions within the chamber. During this stabilization period (lasting about 10 minutes), the damper actuator was activated to set the spring-loaded damper in the closed position, thus blocking air from passing through the rice sample. Once the chamber temperature and RH were stabilized, the combined mass of the drying cylinder and initial mass of the rice sample was measured by activating the load cell actuator. At the end of the weighing cycle, the load cell actuator was deactivated, followed by the deactivation of the damper actuator, which allowed airflow through the drying cylinder to initiate drying (Fig.1).

After a given drying duration, the damper was closed and the weighing procedure repeated. This procedure was repeated every five min for the duration of a drying trial until the change in mass was less than 0.01 g. The drying data were converted to MCs by using the sample mass and MC at the beginning of the drying trial. To facilitate non-linear regression analysis of the drying data, arbitrary k and n values of 0.1 and 0.7, respectively (estimated using data from preliminary tests), and theoretical EMCs estimated using the Modified Chung-Pfost equation, with A, B, and C values from the ASABE standards (2007) were specified for the Page equation (Page, 1949) (Eq. 2). Non-linear regression of the Page equation and the experimental

data was then performed through a series of iterative steps to determine the actual k and n values for each air condition. The asymptotic values of the Page equation were used as the estimation of EMCs for given air temperature and RH conditions.

$$MR = \frac{M - M_{e}}{M_{i} - M_{e}} = e^{-kt^{n}}$$
(2)

where *MR* is the moisture ratio, M_i is the initial moisture content (dry-basis), *M* is the moisture content (dry-basis) after a given drying duration, t, in hours, M_e is the equilibrium moisture content (dry-basis), and *k* and *n* are drying constants.

RESULTS AND DISCUSSION

A total of 72 trials were conducted, comprising four air temperatures, six relative humidities at each temperature, and three replications for each drying condition. Sample plots of MC vs drying duration are shown in Figs. 2 and 3. The drying constants k and n and the corresponding root mean square error (RMSE) determined from the nonlinear regression analysis (JMP. 8.0.1, SAS Institute, Inc., Cary, NC) of the Page equation are shown in Table 1. The low RMSEs (< 0.08) indicated that the Page equation adequately described the experimental data, providing good drying curve estimates.



Figure 2: Fluidized-bed drying data for Cybonnett rice samples dried at 60°C and 7% relative humidity.



Figure 3: Fluidized-bed drying data for Cybonnett rice samples dried at 60°C, and 15, 60, and 75% relative humidities. The solid lines illustrate trends in the drying data.

Table 1: Parameters k and n, and root mean square error (RMSE), a statistical estimate of goodness of fit, determined by nonlinear regression analysis of the Page equation (Eq. 2) with experimental data for Cybonnett rice samples dried in a fluidized-bed system at $60 - 90^{\circ}$ C and 7 - 75% relative humidities. Each value is an average of three replications.

T	Parameter	Relative Humidity (%)						
Temperature (C)		7	15	30	45	60	75	
	k	1.2353	1.7918	1.6589	1.5808	1.4034	0.4429	
60	n	0.8064	0.7087	0.8287	0.7133	0.7853	0.8841	
	RMSE	0.02	0.03	0.04	0.04	0.06	0.09	
70	k	1.4633	1.6334	1.6536	1.7174	1.6449	1.3668	
70	n	0.7195	0.6115	0.7545	0.8933	1.0129	0.7905	
	RMSE	0.04	0.03	0.03	0.03	0.04	0.06	
	k	1.9933	2.1268	3.3679	2.5098	1.8470	2.3987	
80	n	0.6269	0.7497	0.9237	0.8853	1.0492	1.2231	
	RMSE	0.02	0.04	0.04	0.04	0.04	0.05	
	k	2.1530	3.5369	3.3055	5.1912	2.4537	2.6029	
90	n	0.5656	0.7644	0.7220	1.1063	1.0368	1.4975	
	RMSE	0.02	0.03	0.03	0.03	0.03	0.04	

Table 2 shows the EMCs determined as asymptotic values of the Page equation for each temperature and RH combination. There were no significant differences (p-values > 0.05) between replications for all drying conditions. As expected, greater EMCs were measured at greater RHs for the same drying air temperature and lesser EMCs were measured at greater temperatures for the same RH. Similar trends have been reported by Iguaz and Versada, 2007, Chowdhury et al., 2005, Aviara et al., 2004, and Imprasit et al., 2001. The EMC vs RH patterns shown in Figure 4 illustrates a clear effect of temperature and are similar to the Type II isotherms proposed by Brunauer et al. (1940).



Figure 4: Equilibrium moisture contents (%, wet-basis), determined as asymptotic values of the Page equation (Eq. 2) for Cybonnett rice samples dried at $60 - 90^{\circ}$ C and 7 - 75% relative humidities in a fluidized-bed system. Each data point is an average of three replications.

Table 2: Equilibrium moisture contents (%, wet-basis), determined as asymptotic values of the Page equation (Eq. 2) for Cybonnett rice samples dried in a fluidized-bed system at $60 - 90^{\circ}$ C and 7 - 75% relative humidities. Each value is an average of three replications.

	EMC (%)	Relative Humidity (%)						
Temperature (°C)		7	15	30	45	60	75	
60	Mean	6.5	7.3	8.4	9.3	10.9	13.1	
	Std. dev.	0.06	0.08	0.09	0.26	0.25	0.12	
	Mean	5.3	5.8	6.5	8.2	9.5	12.3	
70								
	Std. dev.	0.06	0.03	0.16	0.10	0.04	0.08	
	Mean	4.6	5.6	6.1	7.5	9.4	11.9	
80								
	Std. dev.	0.10	0.07	0.20	0.03	0.14	0.11	
	Mean	3.8	4.7	5.4	6.3	8.0	11.2	
90								
	Std. dev.	0.17	0.03	0.13	0.15	0.15	0.14	

Multiple linear regressions, a statistical analysis used to determine the effect of one or more independent variables on the dependent variable, was used to determine the effect of the temperature and RH and their interactions on EMC. Results showed that the linear effects of both temperature and RH were highly significant (p-values < 0.0001), but there was no significant interaction effect of the two factors on EMC (p-value 0.5602).

The three parameters (A, B, and C) of the Modified Chung-Pfost equation were estimated using nonlinear regression analysis by means of the software JMP. 8.0.1(SAS Institute, Inc., Cary, NC). The ability of the Modified Chung-Pfost equation to accurately estimate EMC was evaluated quantitatively using the root mean square error (RMSE) and coefficient of determination (\mathbb{R}^2). In addition, the pattern of residual plots (i.e., differences between measured and estimated values) was considered as qualitative criterion in evaluating the appropriateness of this model (Chiachung et al., 2001).

Estimates of parameters A, B, and C of the Modified Chung-Pfost equation and the statistical indices used to validate the appropriateness of the model, namely RMSE, R^2 , and patterns of residuals are shown in Table 3. Estimated EMCs from the Modified Chung-Pfost equation with parameters A, B, and C obtained from the ASABE (Standards, 2007) and from a previous study conducted using the same air conditions (Iguaz et al., 2007) were compared to the experimental data. Results showed that the Modified Chung-Pfost equation, with statistically estimated parameters (A, B, and C) gave the best fit with R^2 of 0.94 and RMSE of 0.73.

Table 3: Estimates of coefficients A, B, and C of the Modified Chung-Pfost equation from the ASABE Standards (2007), non-linear regression analysis of experimental data, and Iguaz et al. 2007, and statistical evaluation parameters.

Source	А	В	C	\mathbb{R}^2	RMSE	Pattern of Residuals
ASABE	412.02	0.17528	39.016	0.89	1.04	Random
Iguaz et al. 2007	277.09	0.179	16.912	0.90	0.98	Random
Experiment	438.03	0.3219	-36.994	0.94	0.73	Random

CONCLUSION

The equilibrium moisture contents of long-grain rough rice (Cybonett) were measured using a fluidized-bed drying system. Using A, B, and C values statistically-estimated from the test data, the Modified Chung-Pfost equation was found acceptable for estimating EMCs at temperatures ranging from 60 to 90° C, and RHs 7 to 75%.

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IV. CHAPTER TWO

Drying characteristics and milling quality of rough rice dried in a single-pass incorporating glass transition principles

ABSTRACT

The objective was to study the drying characteristics and milling quality of rough rice subjected to single-pass drying while controlling kernel material states. Samples were harvested at moisture contents ranging from 17.8 to 18.1% from different locations in Arkansas. Drying experiments were conducted at 60, 70, and 80°C and relative humidities ranging from 13 to 83%. High drying air relative humidities (> 63%) maintained both the kernel surface and core in a rubbery state during drying, whereas low drying air relative humidities caused rapid transitioning of the surface layers from a rubbery to a glassy state. Long- and medium-grain cultivars were dried from harvest moisture content to 12.5% moisture content in a single-pass. Immediately after drying, samples were tempered in the drying container or in sealed plastic bags at the drying air temperature for 0, 30, and 60 min, after which they were spread in thin layers and cooled to ambient conditions. The effects of drying air temperatures and relative humidities, tempering conditions, and tempering durations on milling quality was determined. For all drying air temperatures and tempering conditions, milling quality was not significantly different from the controls when the relative humidity of the drying air was maintained above 63% (both the kernel core and surface maintained in a rubbery state during drying) and rice was tempered immediately after drying in sealed plastic bags, at the drying air temperature for at least 60 min. Minimal reduction to milling quality was observed at the low drying-air relative humidities when samples were tempered immediately after drying in sealed plastic bags, at the drying air temperature for at least 60 min; tempering in containers having large headspaces or for shorter

durations failed to reduce the intra-kernel stresses created during drying due to differences in material state between the surface and the core, thereby causing kernel fissuring and subsequent breakage during milling.

Keywords: Drying, Single pass, Glass transition, Relative humidity, Tempering

INTRODUCTION

Drying rough rice to safe storage levels is one of the most important postharvest operations because it influences subsequent handling processes, milling quality, and commercial value of the rice crop (Pan et al., 2011). Minimal reduction in milling quality is often observed when rough rice is dried using unheated air (Basunia and Abe, 2001; Ondier et al., 2010). At low drying air temperatures, thermal expansion, which could lead to kernel fissuring and breakage during subsequent milling, is minimized. However, because of the long durations and limited dryer capacities, the use of low drying air temperatures may not be practical for commercial facilities. To maximize dryer throughput, which is desirable from a logistical standpoint (Siebenmorgen, et al., 2004; Walker and Bakker-Arkema, 1981), high drying air temperatures are used in commercial, cross-flow systems to dry rough rice (Kunze, 1979; Schluterman and Siebenmorgen, 2004). Heated air facilitates shorter drying durations, but has the potential of reducing milling quality if not properly implemented (Kunze, 1979; Schluterman and Siebenmorgen, 2004). Schluterman and Siebenmorgen (2004) described a commercial crossflow dryer operating at drying air temperatures ranging from $40 - 65^{\circ}$ C in multiple passes that lasted approximately 20 - 40 min each. They found that samples located next to the hot-air plenum were more likely to be over dried and of lower milling quality compared to samples located further from the hot-air plenum due to exposure to very high temperatures that caused changes in kernel material states.

Studies have shown that the amount of moisture that can be removed in each drying pass without adversely impacting milling quality is largely dependent on kernel MC (Kunze, 1979; Fan et al., 2000; Cnossen and Siebenmorgen, 2002; Schulterman and Siebenmorgen, 2007). Kunze (1979) showed that rough rice at 20% initial MC can be dried to safe storage levels with minimal reduction to milling quality as long as MC is reduced by a maximum of 1.5 percentage points per hour; greater moisture removal rates resulted in significant reductions in milling quality. Cnossen et al. (2002) and Schulterman and Siebenmorgen (2007) observed similar reduction in milling quality when large amounts of moisture were removed from rice within a short duration. The authors hypothesized that rapid reduction in MC lead to the development of large moisture gradients from the surface to the core of the rice kernel. These moisture gradients in turn produced different material states between the kernel surface and the core, thereby generating stresses within the kernel that resulted in fissuring and breakage during subsequent milling. The material state transformation described by the authors is commonly referred to as glass transition.

Glass transition, one of the most important principles determining polymer applications, is key to understanding high-temperature drying characteristics of starchy grains (Zeleznak and Hoseney, 1987; Schluterman and Siebenmorgen, 2007). At low temperatures or MCs, starch granules behave as a glassy material due to limited molecular motion of the amylose and amylopectin polymer chains. Addition of thermal energy initiates molecular motion within the starch granules, which when excessive, causes the polymer to become viscous, rubbery, and flexible (Zeleznak and Hoseney, 1987). This change in polymer conformation from a glassy to a rubbery material is referred to as glass transition and is accompanied by a large increase in the thermal conductivity (Yang et al., 2002) and expansion coefficient (Perdon et al., 2000), hence

the fast drying rates observed when drying rice at elevated temperatures (Cnossen et al., 2002; Aquerreta et al., 2007). Glass transition is reversible and thus heated polymers can transition from a rubbery to a glassy state when cooled below transition temperatures (Yang et al., 2002) or when MC decreases beyond a certain level (Zeleznak and Hoseney, 1987). Perdon et al. (2000) and Siebenmorgen et al. (2004) developed state diagrams that show increasing glass transition temperatures with decreasing brown rice MC.

Yang and Jia (2004) and Jia et al. (2002a and 2002b) mapped glass transition within a rice kernel during drying and showed that in the rubbery state, the kernel periphery dried much faster and reached equilibrium with the drying air within a shorter duration compared to drying in the glassy state. The rapid loss in moisture was accompanied by transition of the surface layers from a rubbery to a glassy material whereas the core remained as a rubbery material. With extended drying, greater proportions of the kernel periphery transitioned from a rubbery to a glassy material creating tensile and compressive stresses on the surface and core, respectively. The tensile stress along the longitudinal axis peaked and persisted throughout drying, eventually initiating fissuring on the kernel surface (Yang and Jia, 2004; Jia et al., 2002a and 2002b). Schluterman and Siebenmorgen (2007) observed similar material failure and fissure initiation through laboratory experiments designed to produce situations in which large proportions of kernel surface layers transitioned from a rubbery to a glassy material during high-temperature drying.

Considering the great intra-kernel material state differences and the resultant stresses that can be created during high-temperature drying, it is reasoned that by controlling kernel material states during the drying process, the stresses generated within a rice kernel can be minimized. Maintaining the entire kernel as a rubbery material throughout high-temperature drying would

prevent intra-kernel stress development due to differences in material states between the surface and the core of the rice kernel, which has the potential of minimizing fissuring and facilitating maximum moisture reduction within the shortest duration. If realized, this drying approach would conceivably make it possible to dry rough rice from harvest MC to safe storage levels in one pass with minimal reduction to milling quality. Keeping the rice kernel in a rubbery state during drying without allowing surface layers to transition to a glassy state can be implemented by control of the drying air RH. Temperature and RH of the drying air determine the equilibrium moisture content (EMC) of rice exposed to that air, i.e., the greater the RH, the greater the rice EMC at a constant temperature. Based on the state diagram developed by Siebenmorgen et al. (2004), high RHs (> 63%) will maintain the EMCs of the drying air above glass transition whereas low RHs will maintain the EMCs below glass transition (Fig. 1). Shortly after the onset of drying, the MC of the outermost layers of a rice kernel will reach equilibrium with the drying air. If the EMC of the drying air is above glass transition, the outermost layers will remain in a rubbery state throughout the drying process, same as the core, thereby minimizing the internal stresses created by material state differences.



Figure 1: Hypothetical material state differences between the surface and the core of a rice kernel caused by the relative humidity (RH)/equilibrium moisture content (EMC) of the drying air. The glass transition diagram is taken from Siebenmorgen et al., 2004.

Tempering immediately after drying, a common practice in large drying facilities, can be applied to further minimize the internal stresses within a kernel (Cnossen and Siebenmorgen, 2002; Schluterman and Siebenmorgen, 2007; Aquerreta et al., 2007). Tempering allows moisture concentrated in the kernel core to diffuse to the outer layers, rendering less difference in MC and material states between the surface and core. Tempering durations depend on the rate of moisture migration from the core to the surface of rice kernels, which depend on kernel tempering temperatures and MCs, i.e., greater temperatures and MCs result in shorter tempering durations compared to lesser temperatures and MCs (Steffe et al., 1979; Jia et al., 2002a; Cnossen et al., 2002; Aquerreta et al., 2007). Generally, a more uniform intra-kernel moisture distribution, exhibited by reduced fissuring and breakage during subsequent milling, is attained when the tempering duration is prolonged at any temperature (Cnossen et al., 2002).

By incorporating glass transition principles during drying followed by immediate tempering at the end, the main objective of this study was to develop a method that could be used to dry rough rice in a single pass with minimal reduction to milling quality. This approach could make the commercial drying process more efficient by reducing the number of passes and tempering steps.

Layout of the Study

The overall layout of the study is shown in Fig. 2. A series of preliminary experiments were conducted to establish appropriate equipment and procedures for the primary experiment. The first such experiment was conducted to establish drying curves for the three rice lots used in the study. Rice samples were dried from initial MC until EMC was reached for a given drying air condition. From the drying curves, drying durations required to attain 12.5% MC were estimated for each lot and drying air condition.



Figure 2: Experimental layout of the single-pass drying study. The drying characteristics and milling quality of pureline long-grain, Wells, and hybrid, CL XL729, and pureline medium-grain, Jupiter, initially at 17.8 - 18.1 % moisture content was investigated.

The second preliminary experiment was conducted to establish a suitable tempering procedure. Tempering immediately after high-temperature drying is necessary to reduce the moisture gradient developed across the kernel, which may cause material state differences between the kernel core and surface resulting in great intra-kernel stress. The tempering conditions were designed to produce situations whereby the intra-kernel stresses were either adequately or inadequately reduced depending on the tempering durations. Ideally, the duration of tempering must be long enough to allow even distribution of moisture across the kernel, which would render the surface and core of similar material states, thereby reducing the intra-kernel stresses created by material state differences. In addition, tempering temperatures and RHs are critical in high temperature drying processes due to the likelihood of kernel surface transitioning from a rubbery to a glassy state if the RH/EMC of the air within the tempering container and tempering temperature are not maintained above glass transition. Maintaining a high RH/EMC within the tempering container minimizes continued moisture diffusion from the kernel surface during tempering, thereby facilitating rapid MC equilibration throughout the kernel. High RH/EMC also helps minimize material state differences between the surface and the core by maintaining both zones in a rubbery state. The tempering temperatures should be the same as the drying air temperatures or maintained above glass transition to prevent surface layers from transitioning from a rubbery to a glassy state. Tempering at temperatures above glass transition also facilitates faster moisture equilibration across the kernel than tempering at temperatures below glass transition because in the rubbery state, the kernel exhibits greater expansion coefficients and thermal conductivity, resulting in greater moisture diffusivity.

To investigate glass transition of surface layers during tempering, the milling quality of samples tempered at three different conditions was compared. Fissuring and subsequent

reduction in milling quality was expected to be greater in kernels whose surface layers transitioned from a rubbery to a glassy state during tempering compared to kernels that remained in a rubbery state throughout tempering. The first tempering condition studied incorporated a large headspace that allowed moisture migration from the kernel surface into the air within the container throughout tempering; this would cause a large proportion of the surface layers to lose moisture and thus transition from a rubbery to a glassy material during tempering. Such differences in material state were expected to cause substantial intra-kernel stresses that would eventually result in fissuring and subsequent breakage during milling processes. The second condition incorporated a much smaller headspace compared to the first condition, thus limiting moisture loss from the kernel surface, only causing relatively smaller proportions of the kernel surface layers to dry and transition from a rubbery to a glassy material during tempering and thus minimizing fissure formation. Therefore, samples tempered in the limited-headspace container were expected to be of better milling quality compared to samples tempered in the largeheadspace container. The third condition incorporated a sealed and impermeable container with little to no headspace. As a result, the RH within the container was maintained at a high level throughout tempering, thereby preventing moisture loss from the kernel surface and keeping the entire kernel in a rubbery state throughout tempering. Samples tempered in the high RH, air-tight container were expected to of better milling quality than samples tempered in the large- and limited-headspace containers because minimal intra-kernel stress would be generated by the much reduced material state differences between the kernel surface and core.

Wells samples dried in a single pass to 12.5% MC at selected drying air conditions (Fig. 2) were tempered at each of the three tempering conditions immediately after drying i.e., one tempering condition per sample. Subsequent milling qualities were compared among the three

tempering conditions. The sealed/airtight container was selected for the primary experiments because samples tempered therein exhibited the least reduction in milling quality. A detailed description of the preliminary and primary experiments follows.

MATERIALS AND METHODS

Sample collection and preparation

Long- and medium-grain pureline cultivars, Wells and Jupiter, respectively, and a hybrid long-grain cultivar, CL XL729, were harvested from Stuttgart, Arkansas in the fall of 2010 at MCs ranging from 17.8 – 18.1%³. All samples were cleaned (MC[®] Kicker Grain Tester, Mid-Continent Industries, Inc., Newton, KS) and stored in sealed plastic tubs (0.22 m³) at 4°C for two months. Prior to each drying trial, samples were withdrawn from storage, sealed in plastic bags, and allowed to equilibrate to ambient temperature (24°C) overnight. The MC of the rice samples were then measured by drying duplicate, 15-g sub-samples for 24 h in a convection oven (1370 FM, Sheldon Mfg., Inc., Cornelius, OR) maintained at 130°C (Jindal and Siebenmorgen, 1987).

Drying air conditions

A wide range of RHs was selected to maintain the kernel surface either as a glassy or rubbery material during drying and observe the effects of material state differences between the core and the surface on milling quality. Considering the fact that the outermost layers of the kernel surface reach EMC associated with the drying air shortly after the onset of drying, high RHs, which would yield EMCs above glass transition at constant temperature (Fig. 1), were expected to prevent the surface layers from transitioning from a rubbery to a glassy state, thereby minimizing intra-kernel stresses created by material state differences. Low RHs (< 63%), which would yield EMCs below glass transition at constant temperature, were hypothesized to cause

³ Moisture content is expressed on a wet basis unless specified otherwise.
rapid transitioning of the surface layers from a rubbery to a glassy state during drying (Fig. 1), thereby generating the previously-described differential stresses within the kernel that when sufficiently severe, cause fissuring. For this study, rough rice samples were dried at RHs of 13, 23, 33, 43, 53, 63, 73, and 83% and temperatures of 60, 70, and 80°C.High drying air temperatures were selected because of the resultant fast drying rates.

Drying system

A schematic of the system used to conduct the drying experiments is shown in Fig. 3. The apparatus consisted of a controller (ESL 4CA Platinous Temperature and Humidity Chamber, Espec, Hudson, MI) capable of automatically maintaining temperature in the range of -35° C to 150° C ($\pm 0.5^{\circ}$ C) and RH in the range of 6% to 98% (± 1 percentage point) within a 900-L chamber. The air in the chamber was circulated at 0.38 m³s⁻¹to minimize air-condition stratification within the chamber. Air conditions were monitored using a digital temperature and RH probe (Hygro-M2, General Eastern, Woburn, MA). The probe was calibrated using potassium sulfate, sodium chloride, and lithium chloride (Merck, Darmstadt, Germany) prior to the experiments.



Figure 3: Schematic of the fluidized-bed drying system.

A metal cylinder, 20.3 cm (8 in) in diameter and 61.0-cm (24-in) tall, with a perforated floor, a movable lid, and wrapped with 2-mm (0.02-in) thick, ceramic fiber insulation (Zirconia Felt ZY-50, Zircar Zirconia, Inc., Florida, N.Y), was used for establishing the rough rice drying curves. A metal screen, similar in construction to the perforated floor, was fastened to the top opening of the cylinder directly below the movable lid to prevent inadvertent removal of fluidized kernels. This drying apparatus was placed inside the environmental chamber and mounted onto a metal plenum. A 25.4-cm (10-in) diameter centrifugal fan (4C108, Dayton Electric Manufacturing Co., Niles, IL), coupled to a 0.56-kW (0.75-hp), three-phase electric motor (3N443BA, Dayton Electric Manufacturer Co., Niles, IL), was mounted outside the chamber to avoid high-temperature exposure. This fan suctioned air at set temperature and RH from the chamber through a port located in the chamber wall, and then passed the exhaust air through a second port in the chamber wall connected to the plenum beneath the drying cylinder. The desired airflow rate through the drying cylinder was achieved by regulating the electrical frequency of the fan motor using a frequency inverter (AF-300 Mini, GE Fuji Drives USA, Salem, VA), which controlled the motor and fan shaft rotational speed.

A spring-loaded damper constructed in the metal plenum (Fig. 3) controlled airflow direction by either diverting air through the drying cylinder's perforated floor or closing off the perforated floor, allowing the air to empty into the environmental chamber. Opening and closing of the spring-loaded damper was controlled by a linear actuator (damper actuator) (LACT4P, SPAL USA, Ankeney, IA) mounted outside the environmental chamber and connected to the damper by a cable that passed through a port in the chamber ceiling. A second linear actuator (load cell actuator) mounted outside the environmental chamber and directly above the drying cylinder was coupled to a 178-N (40-lb_f) full-bridge, thin-beam load cell (LCL-040, Omega Engineering, Inc., Stanford, CN) that was attached to the drying cylinder via a cable that passed through a second port in the chamber ceiling.

At specified durations, the damper actuator was activated to raise the spring-loaded damper, thereby preventing airflow through the rice sample. The load cell actuator was then activated to suspend the drying cylinder just above the drying apparatus plenum. After a stabilization period, the mass of the drying cylinder and sample was recorded as a millivolt signal from the load cell. Voltage data were converted to masses using a previously-established calibration. Temperature and load cell data were collected using a data logger (21X micrologger, Campbell Scientific, Salt Lake City, UT).

Measurement of airflow rates

Prior to drying trials, fan performance tests were conducted using a metal duct, 15.2 cm (6 in) in diameter and 1.8 m (6 ft) in length, to establish the relationship of the fan motor electrical frequency to the airflow rate of the circulation fan. Airflow rates of 80, 220, 785 cfm/ft² were obtained by setting the fan motor electrical frequency at 10, 20, and 44 Hz, respectively. The average air velocity in the test duct was measured using a hot-wire anemometer (8450-13E-V-STD-NC, Control Co., Friendswood, TX).

The procedure of Subramani et al. (2007) was used to determine minimum fluidization velocity at 60, 70, and 80°C air temperatures. A known mass of rice (1.11 kg) required to attain a 5.1-cm (2-in) grain depth was poured into a transparent cylinder, similar in construction to the previously-described, 61.0-cm (24-in) tall metal cylinder, which allowed for visual observation of rough rice fluidization. The fan was activated and air velocity increased to vigorously fluidize the bed of rice. The air velocity was then slowly decreased until the fluidized bed was reduced to a fixed bed. The pressure drop at different depths across the 5.1-cm (2-in) rice bed was

determined by inserting a static pressure probe (PX270, Omega Engineering Inc., Stamford, CT) from the top of the cylinder as the velocity was being decreased. At minimum fluidization velocity, the pressure drop at different bed depths was constant and the weight of rice was supported by the airflow (Subramani et al., 2007). An airflow rate of 785 cfm/ft² (3.99 m³s⁻¹/m²) was needed to fluidize a 5.1-cm (2-in) rough rice bed in the drying cylinder. This airflow rate was consistent for all rice cultivars and drying conditions and was thus used throughout the study.

Preliminary Experiment 1: Drying curves and durations to attain 12.5% MC

A 1.11-kg (2.45-lb_m) rice sample was placed in the 61-cm (24-in) tall metal cylinder. The drying cylinder was then placed inside the environmental chamber and attached to the load cell actuator. The chamber control system was activated to establish the desired temperature and RH air conditions within the chamber. During this stabilization period, the damper actuator was activated to set the spring-loaded damper in the closed position, thus blocking air from passing through the rice sample. Once the chamber temperature and RH were stabilized, the combined mass of the drying cylinder and initial mass of the rice sample was measured by activating the load cell actuator. At the end of the weighing cycle, the damper actuator was deactivated, which allowed airflow through the drying cylinder to initiate drying (Fig. 3). The weighing procedure was repeated every 5 min for the duration of a drying trial until the change in mass was negligible (less than 0.01 g). The mass data were then converted to MCs based on the initial rough rice sample mass and MC at the start of the drying trial. Drying curves (MC vs drying duration) were plotted for all air conditions specified in Fig. 2 and the durations required to attain 12.5% MC were determined. Each drying run was replicated.

Preliminary Experiment 2: Establishing appropriate tempering conditions

Only the Wells lot was used for these tests. Three tempering conditions were investigated (Fig. 2). Samples were tempered for 0, 30, and 60 min after which they were spread in thin layers and allowed to cool to ambient conditions. In the first tempering condition, rough rice samples were dried and tempered in the 61.0-cm (24-in) tall metal cylinder, which had a large (22-inch) head space that allowed for moisture migration from the kernel surface layers into the air within the cylinder during tempering. In the second condition, samples were dried and tempered in a 15.4-cm (6-in) tall metal cylinder, similar in construction to the 61.0-cm (24-in) tall cylinder, but with a movable lid that fell onto the rough rice samples when the drying airflow was cut off, thus reducing headspace and minimizing moisture migration from the kernel surface during tempering. The second condition was, however, not airtight due to the marginal clearance between the edge of the lid and the inner surface of the cylinder that allowed the lid to slide upwards during drying and downwards during tempering. Limited moisture escape from the drying container during tempering was likely to occur through this clearance. In the third condition, samples dried in the 61.0-cm (24-inch) and 15.4-cm (6-in) cylinders, were manually transferred into 3785-cm³ (1-gal) Ziploc ® plastic bags immediately after drying; air was manually expressed, after which bags were sealed and returned to the drying chamber, where tempering was conducted at the drying air temperature. The transfer of rice from the drying containers into plastic bags immediately after drying was rapid, thus minimizing loss of moisture and energy. At the end of each tempering trial, samples were spread in thin layers and exposed to ambient air at approximately 24°C and 38% RH for 10 min. Each test was replicated. All samples were stored for a week in cold storage (4°C) before milling quality analysis. Post-drying fissuring studies have shown that kernel fissures were mostly visible 24 to 48 h after drying,

thereby justifying the practice of delaying milling quality analysis for more than 48 h after drying (Nguyen and Kunze, 1984; Sharma and Kunze, 1982; Siebenmorgen et al., 2005).

Additional tests were conducted to determine if the actual transfer of rice from the drying containers into plastic bags affected milling quality. Samples from the Wells lot were dried in a single-pass to 12.5% MC using air at 70°C and 13, 23, 33, and 43% RH using the 15.4-cm (6-in) cylinder. At the end of drying, the transfer of rice samples into plastic bags was delayed for 0, 60, 120, and 180 s; during these periods, samples were spread in thin layers and exposed to ambient air (24°C and 38% RH). The samples were then transferred into plastic bags and tempered for 60 min at the drying air temperature. After tempering, samples were again spread in thin layers and exposed to ambient air for 10 min. The samples were then stored for a week in cold storage (4°C) before milling quality analysis. Each test was replicated.

Primary Experiment: Effect of single-pass drying on milling quality

The 15.4-cm (6-in) metal cylinder was used in the primary experiment (Fig. 2). Rough rice samples from all three lots were dried in a single pass to 12.5% MC using air at 60, 70, and 80°C and 13, 23, 33, 43, 53, 63, 73, and 83% RH. At the end of drying, samples were transferred from the drying container into plastic bags and tempered at the drying air temperature for 0, 30, and 60 min. Additional samples from the Wells lot were dried at 70°C and 13, 23, 33, 43, 53, and 63% RH and tempered in plastic bags for 120 min to determine whether extended tempering further improved milling quality. After tempering, samples were spread in thin layers and exposed to ambient air for 10 min. The samples were then stored at 4°C before milling as described previously. Each drying and tempering condition was replicated.

Control samples from each lot were spread in thin layers on screens and gently dried at 26°C and 54% RH from initial MC to 12.5% MC.

Milling analysis

Duplicate, 150-g sub-samples of rough rice, obtained from each sample dried to 12.5% MC, were dehulled using a laboratory huller (Satake Rice Machine, Satake Engineering Co., Ltd., Tokyo, Japan), milled for 30 s using a laboratory mill (McGill #2, Rapsco, Brookshire, TX), and aspirated for 30 s using a seed blower (South Dakota Seed Blower, Seedboro, Chicago, IL). Head rice was then separated from broken kernels using a double-tray sizing machine (Grainman, Grain Machinery MFG, Miami, FL). Head rice was considered as kernels that remained at least three-fourths of the original kernel length after milling (USDA, 2005). Head rice yield (HRY) was calculated as the mass proportion of rough rice that remained as head rice after complete milling.

Statistical analysis

The experimental variables included rice cultivar (pureline and hybrid), cultivar type (medium-grain and long-grain), air conditions (temperature and RH), and tempering conditions and durations. The main effects and interaction of all variables on milling quality were determined using ANOVA (JMP 8.0.1. SAS Institute, Cary, NC). Significant differences between sample means was established using the Tukey test at $\alpha = 0.05$.

RESULTS AND DISCUSSION

Preliminary Experiment 1: Drying curves and durations to attain 12.5% MC

The single-pass drying durations to 12.5% MC for the three cultivars, initially at 17.8 - 18.1% MC and dried at 60, 70, and 80° C and 13 - 83% RH, are shown in Fig. 4. In general, shorter drying durations were observed with increasing temperatures and decreasing RHs because of the resultant low drying air EMC, which formed the driving force for both liquid and vapor transfer from the porous rice kernels (Fortes and Okos, 1981). In addition, drying at the

experimental temperatures, which were above glass transition, resulted in increased kernel volume and moisture diffusivity (Perdon et al., 2000; Siebenmorgen, et al., 2004; Cnossen et al., 2002). Drying durations increased exponentially with increasing RHs at constant drying air temperatures because of the resultant high drying air EMCs.



Figure 4: Drying durations required to attain 12.5% moisture content (MC) for Wells, CL XL729, and Jupiter rice samples, initially at 17.8 – 18.1% MC, dried at $60(\bullet)$, 70 (\Box), and 80°C (\circ) and 13 – 83% relative humidity. The drying durations were estimated from drying curves.

Overall, the single pass drying durations to 12.5% MC were less than durations anecdotally observed when using multi-pass drying methods. The average resident time of rice in a typical commercial cross-flow dryer is approximately 20 – 40 min per pass (Schluterman and Siebenmorgen, 2004). Multiple passes are required to reduce the MC to safe storage levels because only a small amount of moisture (< 3 percentage points) can be removed in each pass to minimize fissure formation and maintain milling quality (Cnossen et al., 2002). The rice is then tempered for extended durations between passes to minimize moisture gradients; generally, more than 24 h are required to dry high-MC rough rice to 12.5% MC. It is, therefore, evident that single-pass drying, even at the high relative humidities, is faster than current multi-pass drying.

Preliminary Experiment 2: Establishing an appropriate tempering condition

The HRYs of Wells samples dried at 70°C and 13% RH in the 61.0-cm (24-in) and 15.4cm (6-in) cylinders, tempered in the cylinders and in sealed plastic bags for 0, 30, and 60 min immediately after drying, and exposed to ambient air for 10 min at the end of tempering, are shown in Fig. 5. As expected, longer tempering durations resulted in greater HRYs for all tempering environments. Also, better milling quality (p-value < 0.05) was observed for samples tempered in sealed plastic bags than for samples tempered in either cylinder. For example, the HRYs of samples dried and tempered in the 15.4-cm (6-in) cylinder for 0, 30, and 60 min were 25.5, 29.1, and 35.5%, respectively, whereas HRYs of samples dried in the same cylinder, but tempered in sealed plastic bags for the same durations were 23.9, 39.5, and 43.9%, respectively. Similar trends were observed for the 61.0-cm (24-in) cylinder.



Figure 5: Head rice yields of Wells samples dried at 70° C and 13% relative humidity to 12.5% moisture content in a single pass in either the 61.0-cm (24-in) or 15.4-cm (6-in) cylinder, and then tempered in the 61.0-cm (large headspace) or 15.4-cm (small headspace) cylinder, or a sealed plastic bag (minimal headspace). Data points are an average of four measurements, i.e., two replications with each replicate head rice yield measured in duplicate. The head rice yield of the control was 51.2%.

Tempering rice in sealed plastic bags was more effective than tempering in either cylinder because at the onset of tempering within the plastic bags, moisture loss from the rice kernels quickly saturated the limited air within the bags; this minimized the vapor pressure difference between the kernel surface and the inter-particle air and prevented further moisture loss from the kernel surface. The high RH within the tempering environment, coupled with the high tempering temperature (70°C) helped maintain both the surface and core in a rubbery state throughout tempering, thereby reducing intra-kernel stresses generated during drying.

Likewise, samples tempered in the 61.0-cm (24-in) cylinder had the lowest milling quality because of the large (22-in) head space that allowed continuous moisture loss from the kernel surface during tempering. The loss of moisture caused a greater proportion of the surface layers to transition from a rubber to a glassy state, increasing the intra-kernel tensile and compressive stress differential generated during drying, and thereby producing fissures (Cnossen and Siebenmorgen, 2002; Jia et al., 2002a; Yang and Jia, 2004). Zhang et al. (2003) observed that tempering rice kernels in the glassy state, even when using high temperatures, did not effectively relieve tensile and compressive stress differential within the kernel and thus only improved milling quality to a limited extent.

Effect of transfer duration from the drying container into plastic bags

The HRYs of samples from the Wells lot, dried at 70°C and 13, 23, 33, and 43% RH to 12.5% MC in a single pass and exposed to ambient air for 0, 60, 120, and 180 s before being placed into plastic bags for tempering are shown in Tab. 1. Loss of milling quality was only significant (p-value < 0.05) for the 180-s exposure. Therefore, milling quality reductions due to the post-drying/tempering container transfer process were significant only for durations greater

than 120 s. The transfer duration for the primary experiment was less than 60 s, hence milling quality reductions due to the post-drying/tempering transfer process were considered negligible.

Table 1: Head rice yields of Wells rice samples dried at 70°C and the indicated relative humidities to 12.5% moisture content in a single pass. Immediately after drying, the samples were exposed to ambient air for 0, 60, 120, and 180 s before tempering in sealed plastic bags for 60 min, after which they were spread in thin layers and exposed to ambient air for 10 min. Head rice yields are an average of four measurements, i.e., two replications with each replicate head rice yield measured in duplicate. Head rice yields are statistically compared within rows. Significant differences between sample means were established using the Tukey test at $\alpha = 0.05$. Values designated by the same superscripted letter are not significantly different.

Relative	Head rice yield, %					
humidity %	0 s	60 s	120 s	180 s		
13	45.9 ^a	44.3 ^{ab}	44.6 ^{ab}	43.4 ^b		
23	45.8 ^a	44.6 ^{ab}	44.1 ^{ab}	43.4 ^b		
33	46.2 ^a	45.0 ^a	45.3 ^a	45.2 ^a		
43	45.9 ^a	45.9 ^a	45.7 ^a	43.9 ^b		

However, in a typical commercial cross-flow drying system (Schluterman and Siebenmorgen, 2004), rice exiting a dryer is normally exposed to ambient air as it is conveyed by unloading augers and bucket elevators to tempering/storage bins. The conveying process during which kernels are exposed to ambient air, is most likely longer than 120 s, and could be a cause for some milling quality reduction.

Primary Experiment: Effect of single-pass drying on milling quality

Milling quality of pureline, long-grain, Wells, hybrid, long-grain, CL XL729, and pureline, medium-grain, Jupiter, cultivars dried in a single pass at 60, 70 and 80°C and 13, 23, 33, 43, 53, 63, 73, and 83% RH to 12.5% MC followed by immediate tempering in sealed plastic bags for 0, 30, and 60 min after which samples were spread in a thin layer and exposed to ambient air for 10 min to allow for cooling are shown in Figs. 6 - 10. Milling quality improved with increasing RHs for all drying air temperatures. The trend was more prominent for samples tempered for 0 and 30 min compared to samples tempered for 60 min. The lesser HRYs observed at 13 – 53% RH compared to 53 – 83% RH may be explained by intra-kernel stresses resulting from material state differences between the surface and core. Shortly after coming into contact with the drying air, the kernel reached drying air temperatures and transitioned from a glassy to a rubbery state. As indicated by Yang and Jia (2004), the MC of the outermost layers reduced rapidly to the low EMC associated with the drying air conditions (Fig. 1 and Table 2), whereas the core remained at a relatively high MC. A moisture gradient developed from the core to the surface of the kernel, which increased in magnitude as drying progressed due to increasing proportions of the surface layers reaching EMC associated with the drying air (Yang and Jia, 2004). The low surface MC caused surface layers to transition from a rubbery to a glassy state while the core remained rubbery, creating material state differences within the kernel. The intrakernel stresses resulting from the material state differences between the surface and the core caused massive kernel fissuring when rice was not tempered immediately after drying, hence the low milling quality observed after 0 min of tempering. Tempering for at least 30 min resulted in significant improvement in milling quality, which indicates that the 30-min tempering duration, though inadequate, helped reduce the intra-kernel stress differential. Tempering for at least 60 min resulted in much better milling quality compared to tempering for 0 and 30 min, which showed that the intra-kernel stresses were greatly reduced within this duration. However, the milling quality of the samples dried at the low drying air RHs was still less than the control even after 120 min of tempering (Fig. 7), which shows that the intra-kernel stress created by material state differences between the surface and the core could not be completely eliminated. Table 2: Equilibrium moisture contents of rough rice estimated using the Modified Chung-Pfost Equation for temperatures of 60, 70, and 80° C and 13 - 83% relative humidities. The empirical constants A, B, and C of the equation were estimated by Ondier and Siebenmorgen (2010) for high-temperature conditions.

	Equilibrium Moisture Content, % w.b							
Temperature	13%	23%	33%	43%	53%	63%	73%	83%
(°C)								
60	6.5	7.4	8.1	8.8	9.6	10.4	11.3	12.6
70	5.5	6.4	7.2	7.9	8.6	9.4	10.4	11.7
80	4.8	5.7	6.4	7.2	7.9	8.8	9.7	11.1



Figure 6: Head rice yields of Wells rice samples dried at 60° C and 13 - 83% relative humidity to 12.5% moisture content in a single pass. The samples were dried in the 15.4-cm (6-in) cylinder and immediately tempered in plastic bags for 0 (•), 30 (□), and 60 min (°). The control samples (----) were gently dried at 26°C and 54% relative humidity to 12.5% moisture content. Data points are an average of four measurements, i.e., two replications with each replicate head rice yield measured in duplicate.



Figure 7: Head rice yields of Wells rice samples dried at 70°C and 13 - 83% relative humidity to 12.5% moisture content in a single pass. The samples were dried in the 15.4-cm (6-in) cylinder and immediately tempered in plastic bags for 0 (•), 30 (\Box), and 60 min (\circ). Additional samples dried at 70°C and 13 – 43% RH were tempered for 120 min (\blacksquare). The control samples (----) were gently dried at 26°C and 54% relative humidity to 12.5% moisture content. Data points are an average of four measurements, i.e., two replications with each replicate head rice yield measured in duplicate.



Figure 8: Head rice yields of Wells rice samples dried at 80°C and 13 - 83% relative humidity to 12.5% moisture content in a single pass. The samples were dried in the 15.4-cm (6-in) cylinder and immediately tempered in plastic bags for 0 (\bullet), 30 (\Box), and 60 min (\circ). The control samples (----) were gently dried at 26°C and 54% relative humidity to 12.5% moisture content. Data points are an average of four measurements, i.e., two replications with each replicate head rice yield measured in duplicate.



Figure 9: Head rice yields of Jupiter rice samples dried at 60° C and 13 - 83% relative humidity to 12.5% moisture content in a single pass. The samples were dried in the 15.4-cm (6-in) cylinder and immediately tempered in plastic bags for 0 (•), 30 (□), and 60 min (°). The control samples (----) were gently dried at 26° C and 54% relative humidity to 12.5% moisture content. Data points are an average of four measurements, i.e., two replications with each replicate head rice yield measured in duplicate.



Figure 10: Head rice yields of CL XL729 rice samples dried at 60° C and 13 - 83% relative humidity to 12.5% moisture content in a single pass. The samples were dried in the 15.4-cm (6-in) cylinder and immediately tempered in plastic bags for 0 (•), 30 (□), and 60 min (°). The control samples (----) were gently dried at 26°C and 54% relative humidity to 12.5% moisture content. Data points are an average of four measurements, i.e., two replications with each replicate head rice yield measured in duplicate.

The increasing HRYs observed when RH was increased from 53 to 83% were, likewise, due to the greater EMC of the drying air (Fig. 1 and Table 2), which reduced material state differences between the kernel surface and core, thereby reducing intra-kernel stress differential. As previously described, the outermost layers of the rice kernel reaches EMC associated with the drying air conditions shortly after the onset of drying. Because increasing rice EMCs are associated with the increasing drying air RHs, the material state differences between the kernel core and surface were relatively reduced at high drying air RHs compared to when drying at lower RHs, hence less intra-kernel stress differential was created. At RHs greater than 63%, it is highly likely that both the surface and the core were maintained in a rubbery state during drying (Fig. 1), greatly reducing intra-kernel stress created by differences in material state and thus resulting in milling quality that was not significantly different from the control after 60 min of tempering (Yang and Jia, 2004). Similar results have been reported by Schulman et al. (1993) and Cnossen and Siebenmorgen (2002) who observed improved milling quality in samples dried using high RH/EMC air and tempered for at least 60 min.

Medium-grain vs long-grain kernel fissuring susceptibility

The medium-grain cultivar, Jupiter, whose features include a shorter, thicker kernel, was more susceptible to fissuring after 0 and 30 min of tempering (Fig. 9) compared to the long-grain cultivars, Wells and CL XL729 (Figs. 6 and 10) e.g. for drying air conditions of 60°C and 13, 23, and 33% RH, and a tempering duration of 30 min, the HRYs of Wells samples were 46.2, 45.1, and 46.3%, respectively, the HRYs of CL XL729 samples were 37.8, 42.5, and 47.3%, respectively, whereas the HRYs of Jupiter samples were 33.5, 32.4, and 35.4%, respectively. Studies have consistently shown that medium-grain kernels are more susceptible to fissuring than long-grain kernels (Jindal and Siebenmorgen, 1994; Fan et al., 2000; Siebenmorgen et al., 2005).

Sun et al (2001) reported that medium-grain kernels showed greater non-uniform kernel length and thickness shrinkage during drying compared to long-grain kernels, which could be the ultimate reason that medium-grain kernels are more susceptible to fissuring than long-grain kernels.

CONCLUSION

High drying air temperatures (> 60° C) can be used to dry rough rice in a single pass with minimal reduction to milling quality provided samples are properly tempered for at least 1 h immediately after drying. Alternatively, maintaining a high drying air RH will keep the kernel surface and core in a rubbery state, thereby minimizing the intra-kernel stress caused by differences in material properties, and thus prevent fissuring.

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V. CHAPTER THREE

Physicochemical properties of rough rice dried in a single pass using high temperatures ABSTRACT

This study evaluated the physicochemical properties of high-temperature, single-passdried rough rice. Pureline cultivar Wells (long-grain) and Jupiter (medium-grain) and hybrid cultivar CL XL729 (long-grain), at initial moisture contents of 17.9 – 18.1%⁴ were dried in a single-pass to approximately 12.5% moisture content using drying air temperatures of 60, 70, and 80°C and relative humidities of 13 to 83%. Immediately after drying, the samples were tempered for one hour at the drying air temperatures in sealed plastic bags. Color, degree of milling, pasting viscosity, and thermal properties of the milled rice were evaluated. Results showed that color, degree of milling, and thermal properties were not affected by drying air temperatures and relative humidities. However, peak, final, and breakdown viscosities increased with increasing drying air temperatures and relative humidities in all three cultivars. **Keywords:** Single-pass, Rice, Fluidized-bed drying, Pasting viscosity, Thermal properties

INTRODUCTION

The rice industry continuously strives to develop faster drying methods to handle the increasing crop influx resulting from high yielding cultivars and rapid harvesting and transportation systems (Truitt and Siebenmorgen, 2006). Drying rough rice in a single pass at high temperatures (> 60° C) by incorporating glass transition principles in which kernel material states are controlled during drying, coupled with proper tempering for at least one hour immediately after drying, has the potential to increase dryer throughput with minimal reduction to milling quality (Ondier and Siebenmorgen, 2012; Cnossen and Siebenmorgen, 2002). In

⁴ Moisture content is expressed on a wet basis unless specified otherwise.

addition, drying with high air temperatures has the potential of minimizing the deleterious activity of enzymes such as amylases, proteases, and lipases, which are sensitive to heating (Halick and Kelly, 1959; Tani et al., 1964; Iwasaki et al., 1965; Iwasaki and Tani, 1966; Dhaliwal et al., 1991).

Drying rough rice using high temperatures can, however, influence milled rice functionality, affecting parameters such as color (Dillahunty et al., 2001; Prachayawarakorn et al., 2005; Soponronnarit et al., 1999), cooking properties (Iwasaki and Tani, 1966; Tirawanichakul et al., 2004; Wiset et al., 2001), and eating quality (Champagne et al., 1998; Inprasit and Noomhorm, 2001). For example, Dillahunty et al. (2001) reported increasing milled rice discoloration with increasing rough rice drying temperatures (>50°C) and durations; Wiset et al. (2001) observed greater water absorption in rough rice samples dried at temperatures of 85 -90°C compared to samples dried at ambient temperatures; Iwasaki and Tani 1966 showed that heating rough rice to high temperatures (>60°C) for 1 h resulted in considerable changes in cooked rice texture and off-flavor development, subsequently recommending temperatures below 50°C as the safe limit of heating during artificial drying; and Champagne et al. (1998) reported greater values of cooked-rice cohesiveness for samples dried at 60°C than for samples dried at 18°C and presumed the increase to result from fissures developed from the harsh drying conditions, which increased water absorption during cooking.

Though results are conflicting, studies have also shown that drying conditions influence milled rice flour physicochemical properties such as pasting viscosity and thermal properties, which are strongly correlated with end-use product quality (Dhaliwal et al., 1991; Pearce et al., 2001; Tran et al., 2001; Dillahunty et al., 2001; Patindol et al., 2003; Wang et al., 2004; Dang and Copeland, 2004). For example, Dhaliwal et al. (1991) showed that rough rice samples stored

without drying had greater gelatinization and peak viscosity than samples stored after drying; Dillahunty et al. (2001) showed that exposing rice to high temperatures (60°C) for extended durations (48 h) decreased peak viscosity; and Patindol et al. (2003) observed greater pasting viscosity in medium-grain samples dried at 40 and 60°C compared to samples dried at 20°C.

Though single-pass drying at high temperatures has the potential of maximizing dryer capacity by increasing rough rice drying rates and dryer throughput, the effects of high drying-air temperatures on end-use product functionality need to be investigated. The objective of this study was to determine the effect of single-pass, high-temperature drying of rough rice on milled rice appearance and functionality. Color, degree of milling, pasting viscosity profiles, and thermal properties were evaluated.

MATERIALS AND METHODS

Sample collection and preparation

Long- and medium-grain pureline cultivars, Wells and Jupiter, respectively, and a hybrid long-grain cultivar, CL XL729, were harvested from Stuttgart, Arkansas in the fall of 2010 at MCs ranging from 17.8 – 18.1%. All samples were cleaned (MC[®] Kicker Grain Tester, Mid-Continent Industries, Inc., Newton, KS) and stored in sealed plastic tubs (0.22 m³) at 4°C for two months. Prior to each drying trial, samples were withdrawn from storage, sealed in plastic bags, and allowed to equilibrate to room temperature (24°C) overnight. The MC of the rice samples were then measured by drying duplicate, 15-g samples for 24 h in a convection oven (1370 FM, Sheldon Mfg., Inc., Cornelius, OR) maintained at 130°C (Jindal and Siebenmorgen, 1987).

Drying conditions

The fluidized-bed system described by Ondier et al 2010 was used for the drying experiments. Five hundred-g rough rice samples were dried in a single pass at 60, 70, and 80°C

and 13, 23, 33, 43, 53, 63, 73, and 83% RH from initial MC to approximately 12.5% MC. Immediately after drying, samples were tempered in sealed plastic bags, at the drying air temperature, for one hour to allow moisture equilibration within the rice kernels, thereby minimizing fissuring upon exposure to cooling air (Nguyen and Kunze, 1984; Cnossen and Siebenmorgen, 2002). Each drying trial was replicated. Control samples from each cultivar lot were spread on screens in thin layers and gently dried to 12.5% MC in an environment where temperature and RH were maintained at 26°C and 54%, respectively, using a conditioning system (Model AA-558, Parameter Generation & Control, Inc., Black Mountain, NC). After drying, all samples were stored at 4°C for approximately four months before quality analyses.

Milling analyses

Duplicate, 150-g sub-samples of rough rice, obtained from each sample dried to the desired 12.5% MC, were dehulled using a laboratory huller (Satake Rice Machine, Satake Engineering Co., Ltd., Tokyo, Japan), milled for 30 s using a laboratory mill (McGill #2, Rapscore, Brookshire, TX), and aspirated for 30 s using a seed blower (South Dakota Seed Blower, Seedboro, Chicago, IL) to remove loose bran particles from the surface of rice kernels. Head rice was then separated from broken kernels using a double-tray sizing machine (Grainman, Grain Machinery MFG, Miami, FL). Head rice was considered as milled-rice kernels that remained at least three-fourths of the original kernel length (USDA, 2005).

Color

The whiteness and yellowness of duplicate, 15-g head rice sub-samples were measured using a Commission Internationale de l'Eclairage system L* and b* color scale, respectively, with a colorimeter (ColorfLex, Hunter Association Laboratory, Reston, VA). The instrument was calibrated before testing using black and white plates supplied by the manufacturer. Samples

were placed in a clear plastic cup and covered with a black plastic cup prior to color measurement to avoid interference from ambient light. After taking the first color reading, the sample cup was rotated approximately 180° and a second reading of the same sample was taken. Both readings were averaged to obtain one whiteness (L^{*}) and yellowness (b^{*}) measurement. The color measurements were reflective indices of the sample surface, i.e., the greater the L* value, the whiter the milled rice and the greater the b* value, the more yellow the milled rice.

Degree of milling

Surface lipid content was measured as an indication of the degree of milling. Surface lipids were extracted using a Soxtec extractor (Avanti 2055, Foss North America, Eden Prairie, MN) according to the method of Matsler and Siebenmorgen (2005). Duplicate, head rice samples (4 to 5 g) were pre-dried at 100°C for 1 h, boiled in petroleum ether for 20 min, and rinsed with petroleum ether condensate for 30 min. Extraction cups containing the extracted lipids were dried at 100°C for 30 min to remove residual petroleum ether and weighed. Surface lipid content was determined as the mass ratio of lipids extracted from the surface of kernels to the original milled rice sample mass.

Pasting viscosity profiles

To determine pasting viscosity profiles, duplicate, 20-g head rice sub-samples were ground into flour using a cyclone mill with a 0.5-mm sieve (model 2511, Udy Corp., Fort Collins, CO). The MC of the flour was determined by drying duplicate, 5-g samples in a convection oven at 130°C for 1 h (Jindal and Siebenmorgen, 1987). Flour samples were prepared for viscosity analysis by mixing 3 ± 0.01 g of flour (at approximately 12% MC) with 25 ± 0.05 ml deionized water. Water corrections were made to account for the samples being above or below 12% MC. Peak and final viscosities of the rice flour were determined using a Rapid Visco Analyzer TM (RVA) (model 4, Newport Scientific, Warriewood, NSW, Australia). Setback viscosity was calculated as the difference between final and peak viscosities. The RVA was set up on a 12.5 min runtime (1.5 min at 50°C, heating to 95°C at 12°C/min, 2.5 min at 95°C, cooling to 50°C at 12°C/min, and held for 1 min at 50°C) according to AACC Methods, 1996. Peak and final viscosities were recorded in centipoise (1 RVA unit = 10 cP).

Thermal properties

Differential scanning calorimetry (DSC) was used to measure thermal properties of rice flour. Duplicate flour samples (4 + 0.1 mg) were weighed into aluminum pans, and hydrated with 8 μ L of deionized water using a micro-syringe. The pans were hermetically sealed and allowed to equilibrate for 12 h. Differential scanning calorimetry was conducted using a calorimeter (Pyris 1, Perkin Elmer, Norwalk, CT) at a scanning rate of 10°C/min from 25°C to 120°C with an empty pan as the reference. The onset (T_o), peak (T_p), and conclusion (T_c) gelatinization temperatures and gelatinization enthalpy of the rice flour were obtained from each DSC thermogram generated by system software (Pyris series version 9.1, Perkin Elmer).

Statistical Analysis

The experimental variables included rice cultivar (pureline and hybrid), kernel type (medium-grain and long-grain), and drying conditions of temperature and RH. The main and interactions effects of all variables on milled rice physicochemical properties were determined using Analysis of Variance (JMP 9.0.1., SAS Institute, Cary, NC). Means were compared using Tukey significance test at $\alpha = 0.05$.

RESULTS AND DISCUSSION

Color

Whiteness, expressed as L* values, and yellowness, expressed as b* values, of Wells, Jupiter, and CL XL729 rice samples dried in a single pass at temperatures of 60, 70, and 80°C and RHs of 13 - 83%, and then tempered at the drying air temperature for 1 h immediately after drying are shown in Table 1. No significant differences were observed between the whiteness and yellowness of controls and experimental samples dried at increasing temperatures and RHs (p-value > 0.05). It is important to note that at constant temperature, samples dried at 83% RH were exposed to the drying air conditions for longer durations compared to samples dried at 13% RH, e.g., the drying durations to 12.5% MC for Wells samples initially at 18.1% MC and dried at 13% RH and 60, 70, and 80°C were 24, 22, and 14 min, respectively, whereas the durations at 83% RH were 170, 120, and 105 min, respectively. The results therefore, show that rice can be dried at high temperatures for almost 3 h without a significant decrease in whiteness or increase in yellowness. Table 1: Whiteness, expressed as L* values, and yellowness, expressed as b* values, of Wells, Jupiter, and CL XL729 rice samples dried in a single pass at 60, 70, and 80°C and 13 to 83% relative humidity to the desired 12.5% moisture content. Control samples were dried at 26°C and 54% relative humidity to 12.5% moisture content. Each value is an average of duplicate measurements.

Temperature	Relative humidity	Wells		Jupiter		CL XL729	
(°C)	(%)	L*	b*	L*	b*	L*	b*
60	13	73.7 ^a	14.6 ^b	71.6 ^c	16.2 ^d	71.4 ^e	15.0 ^f
	23	73.9 ^a	15.2^{b}	72.6 ^c	15.9 ^d	71.1 ^e	$14.7^{\rm f}$
	33	73.7 ^a	14.8^{b}	72.6 ^c	15.9 ^d	70.7 ^e	$15.4^{\rm f}$
	43	73.0 ^a	14.3 ^b	72.7 ^c	16.2 ^d	71.0 ^e	15.1 ^f
	53	73.4 ^a	14.8 ^b	72.1 ^c	16.2 ^d	71.6 ^e	$15.4^{\rm f}$
	63	74.0^{a}	14.8^{b}	72.7 ^c	16.3 ^d	70.9 ^e	14.4^{f}
	73	73.5 ^a	15.5 ^b	72.5 ^c	16.2^{d}	70.8 ^e	$15.0^{\rm f}$
	83	74.2 ^a	15.0^{b}	72.5 ^c	15.7 ^d	71.1 ^e	14.7 ^f
70	13	74.1 ^a	14.9 ^b	72.3 ^c	16.3 ^d	71.5 ^e	15.0 ^f
	23	74.4 ^a	14.7^{b}	72.7 ^c	17.0^{d}	71.1 ^e	15.0 ^f
	33	74.4 ^a	15.4 ^b	72.8 ^c	16.2^{d}	70.7 ^e	14.6 ^f
	43	73.5 ^a	14.8 ^b	72.6 ^c	16.4 ^d	71.0 ^e	14.9 ^f
	53	74.3 ^a	15.1 ^b	72.4 ^c	16.6 ^d	70.3 ^e	15.2 ^f
	63	74.3 ^a	15.1 ^b	72.1 ^c	16.3 ^d	70.7 ^e	15.1 ^f
	73	74.4 ^a	15.4 ^b	72.4 ^c	16.3 ^d	70.5 ^e	15.2 ^f
	83	74.5 ^a	14.8 ^b	72.6 ^c	17.2 ^d	69.7 ^e	15.8 ^f
80	13	74.0 ^a	15.1 ^b	71.3 ^c	17.0^{d}	71.1 ^e	15.0 ^f
	23	74.5 ^a	15.0^{b}	73.1 ^c	16.3 ^d	70.9 ^e	15.5 ^f
	33	74.4 ^a	15.3 ^b	72.6 ^c	16.6^{d}	70.3 ^e	15.5 ^f
	43	73.5 ^a	15.8 ^b	72.2 ^c	16.6 ^d	70.7 ^e	15.6 ^f
	53	73.9 ^a	15.8 ^b	73.2 ^c	16.9 ^d	70.3 ^e	15.0^{f}
	63	74.9 ^a	15.9 ^b	72.6 ^c	17.2^{d}	70.2 ^e	15.4 ^f
	73	74.5 ^a	15.4 ^b	72.7 ^c	17.2^{d}	69.8 ^e	16.2^{f}
	83	73.7 ^a	16.5^{b}	72.3 ^c	17.6^{d}	70.5 ^e	$16.2^{\rm f}$
Control		73.1 ^a	15.8 ^b	72.2 ^c	16.7 ^d	71.8 ^e	15.4 ^f

* Values are compared within columns.

*Values designated by the same alphabetical letter are not significantly different (p-value 0.05).
Patindol et al. (2003) observed similar trends where whiteness (L* values) of mediumgrain, Bengal, and long-grain, Cypress cultivars, dried at 20, 40, and 60°C to 12.5% MC in a single pass were not significantly different (p-value > 0.05). Some studies have reported increased yellowness with exposure to temperatures ranging from $45-50^{\circ}$ C (Dillahunty et al., 2001; Bunyawanichakul et al., 2005; Ambardekar and Siebenmorgen, 2012). The yellowing could have been caused by increasing enzymatic activity at temperatures less than 50° C (Soponronnarit et al., 1990). Most enzymes would have been inactivated at the current experimental temperatures of 60, 70, and 80°C, thereby preventing enzymatic reactions known to decrease whiteness and increase yellowness. Increased solubility of the reactive species when high MC rough rice is exposed to high temperatures has also been proposed as a possible cause for decreased whiteness and increased yellowness. This preposition is supported by findings from Ambardekar and Siebenmorgen (2012) who showed that rice yellowing was greater in high-MC samples exposed to temperatures of 60 to 80°C compared to low-MC samples exposed to the same temperatures and exposure durations. Belefant-Miller et al. (2005) reported similar findings in an induced-yellowing study where milled samples incubated at 78°C for 114 h in test tubes with 10 μ L of water showed greater yellowing than samples incubated in dry test tubes for the same duration. The authors concluded that MC was an important factor in inducing yellowing in rice exposed to elevated temperatures. Ambardekar and Siebenmorgen 2012 also showed that exposure durations of 4 h at 60°C and 30 min at 80°C were necessary to induce significant yellowing in high-MC (> 18%) samples. In the current study, drying rough rice in a single pass at high temperatures quickly reduced MC within the first few minutes of exposure, thereby limiting the mobility and interaction of the reactive species; this may have prevented yellowing or browning reactions, thereby maintaining milled rice color.

Degree of milling

Degree of milling values, expressed as surface lipid content (SLC), of rice samples dried at the experimental conditions, are shown in Figure 1. Results show that increasing drying air temperatures from 60 to 80° C and RHs from 13 to 83% did not affect bran removal rates in either the pureline or hybrid cultivars (p-values > 0.05). However, the degree of milling values of the experimental and control samples were significantly different, i.e., the SLC of hybrid, CL XL729, samples were significantly greater (p < 0.05) than SLC of the control, whereas SLCs of the purelines, Jupiter and Wells, samples were significantly lesser (p < 0.05) than SLCs of the controls. Generally, hybrids have a lower SLC (Siebenmorgen et al., 2006) and greater degree of milling (Lanning and Siebenmorgen, 2011) than pureline cultivars for comparable milling durations, i.e., hybrids have a smaller bran layer compared to pureline cultivars. The hightemperature drying conditions may have increased adherence of the thin bran layer to the endosperm of the hybrid cultivar, but loosened the outer layers of the relatively thick bran layer of the pureline cultivars.



Figure 1: Degree of milling, expressed as surface lipid content, of Wells, Jupiter, and CL XL729 rice samples dried in a single pass at 60 (\bullet), 70 (\Box), and 80°C (\circ) and 13 to 83% relative humidity to the desired 12.5% moisture content. Control samples (----) were dried at 26°C and 54% RH to 12.5% moisture content. Each value is an average of duplicate measurements.

Pasting Property

At constant temperature, increasing drying air RHs did not have a significant effect on peak and final viscosities of rice flour from all three cultivar lots. However, peak and final viscosities of all three cultivar lots were significantly greater (p-value < 0.05) than the controls at 70 and 80°C (Fig. 2). Dhaliwal et al. (1999) reported similar increases in peak and final viscosities with increasing drying air temperatures. Ambardekar and Siebenmorgen (2012) reported similar increases in peak and final viscosities for samples exposed to elevated temperatures, attributing the increase to modification of the starch granule structures. High peak and final viscosities are associated with increased cooked rice cohesiveness (Champagne et al., 1998) due to increasing water binding capacity of the starch granules and subsequent disintegration during cooking (Wang et al., 2004; Ruby and Elsie, 1960).

Similar to peak and final viscosities, breakdown viscosity of all three rice lots increased with increasing drying air temperatures, e.g., the breakdown viscosities of Wells samples dried at 60, 70, and 80°C were 1156, 1227, 1354 cP, respectively; the breakdown viscosity of the control was 1104 cP. Dang and Copeland (2004) reported similar findings showing that postharvest processes such as drying affected the rate at which starch swell and breakdown causing granular components to leach out of the rice kernel during exposure to high temperature and shear. The authors hypothesized that high drying air temperatures caused cross-linking of starch granules with unfolded proteins or lipids, making the granules resistant to breakdown, hence the high breakdown viscosity.

Setback viscosity, which is the difference in viscosity between the minimum (trough) and the final viscosity value at the end of the test due to retrogradation of amylose and amylopectin during cooling of the gelatinized starch, decreased with increasing drying air temperatures and

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was notably lower than the controls in the both pureline cultivars, Wells and Jupiter (Figure 2). However, the setback viscosities of the hybrid, CL XL729, samples were not affected by increasing drying air temperatures. Ambardekar and Siebenmorgen (2012) observed similar results where samples from CL XL729 had greater setback viscosities than samples from Wells after exposure to elevated temperatures. The authors attributed the difference in setback viscosities to inherent differences between the starch structures of the two cultivars that affected their differential ability to retrograde after cooling.



Figure 2: Peak, final, and setback viscosities of Wells, Jupiter, and CL XL729 rice samples dried in a single pass at 60 (\bullet), 70 (\Box), and 80°C (\circ) and 13 to 83% relative humidity to the desired 12.5% moisture content. Control samples (----) were dried at 26°C and 54% RH to 12.5% moisture content. Each value is an average of duplicate measurements.

In summary, the changes in pasting viscosity profiles observed in the experimental samples may be attributed to chemical and structural changes occurring within the rice kernels when exposed to high drying air temperatures. Generally, starch and not protein or other constituents has the greatest influence on the pasting viscosity profile of rice flour (Patindol et al., 2003). However, protein, which is the second largest component in rice flour, plays a significant role in pasting property because starch granules are encased in the protein matrix, causing the disulfide bonds to restrict granule swelling during gelatinization (Ruby and Elsie, 1960; Hamaker and Griffin, 1993; Baxter et al., 2010). In addition, soluble proteins such as albumin, which constitutes approximately 12% of milled rice protein (Chandi and Sogi, 2007), may obstruct water access to the starch granule through preferential binding of starch hydrophilic region, thus reducing water uptake and limiting starch granule swelling (Baxter et al., 2010; Evers et al., 1999; Bean and Lockhart, 2001). Given that the denaturation temperatures of rice protein (albumin, globulin, and glutelin) ranges between 70 to 80°C (Ju et al., 2001), it is highly likely that the drying air temperatures of 70 and 80° C, which were used in the current study, disrupted the disulfide bonds, thus causing the starch-granule to swell to a larger size (Hamaker and Griffin, 1993). In addition, the amylose and amylopectin chains of the starch granule may have interacted with the unfolded structures of the denatured protein (Dang and Copeland, 2004), forming complexes that further increased the pasting viscosity profile of the experimental samples. Patindol et al. (2003) found drying air temperatures of $40 - 60^{\circ}$ C to have no apparent effect on the pasting viscosity profile of pure/isolated starches, which supports the role of protein-starch complex on pasting viscosity.

Thermal Properties

The thermal properties (onset, peak, and gelatinization enthalpy) of the Wells, Jupiter, and CL XL729 samples dried at increasing temperatures and RHs were not significantly different (p > 0.05) from the controls (Table 2). Patindol et al. (2003) reported similar findings showing that rough rice drying conditions had no apparent effect on thermal properties of rice flour. Ambardekar and Siebenmorgen (2012) reported similar results for rough rice samples exposed to elevated temperatures for extended durations.

Table 2: Onset (T_o), peak (T_p), and conclusion (T_c) gelatinization temperatures ($^{\circ}$ C) and gelatinization enthalpy (E) (J/kg) of rice flour from Wells, Jupiter, and CL XL729 rice samples dried in a single pass at 60, 70, and 80°C and 13 to 83% relative humidity (RH) to the desired 12.5% moisture content. Control samples were dried at 26°C and 54% RH to 12.5% moisture content. Each value is an average of duplicate measurements.

Temp	RH	Wells			Jupiter				CL XL729				
(°C)	(%)	To	Tp	Tc	Ε	To	Tp	Tc	Ε	To	Tp	T _c	E
60	13	75.9 ^a	80.6 b	86.5 c	8.9 ^d	64.3 e	70.2 ^f	77.1 ^g	5.4 ^h	75.0 ⁱ	79.6 ^j	87.0 ^k	8.6 ¹
	53	76.1 a	84.0 b	84.2 c	9.3 ^d	64.2 e	69.9 ^f	76.8 g	7.0 ^h	74.6 ⁱ	79.3 ^j	86.3 ^k	7.6 ¹
	83	74.7 ^a	80.2 b	87.7 c	9.1 ^d	63.9 e	69.8 ^f	76.1 g	6.1 ^h	73.3 ⁱ	78.9 ^j	86.8 ^k	9.4 ¹
70	13	75.7 ^a	80.4 b	86.1 c	9.6 ^d	64.1 e	69.9 ^f	76.6 g	6.3 ^h	73.8 ⁱ	79.0 ^j	86.7 ^k	8.2 ¹
	53	75.7 ^a	80.3 b	85.5 c	8.8 ^d	66.0 e	72.5 ^f	78.8 g	7.0 ^h	74.4 ⁱ	80.1 ^j	86.6 ^k	8.2 ¹
	83	75.7 ^a	80.6 ^b	86.5 c	9.7 ^d	64.9 e	70.2 ^f	76.7 ^g	7.0 ^h	74.6 ⁱ	79.7 ^j	87.1 ^k	8.2 ¹
80	13	76.5 ^a	81.3 b	87.2 c	8.9 ^d	64.3 e	70.0 ^f	76.1 g	6.6 ^h	74.4 ⁱ	79.8 ^j	87.0 ^k	8.5 ¹
	53	75.7 ^a	80.3 b	85.9 c	9.1 ^d	64.0 e	69.6 ^f	76.2 g	7.0 ^h	74.2 ⁱ	79.1 ^j	83.8 ^k	9.1 ¹
	83	75.7 ^a	80.4 b	86.2 c	8.7 ^d	63.7 e	69.3 ^f	75.7 ^g	7.3 ^h	74.4 ⁱ	79.5 ^j	87.1 ^k	9.2 ¹
Control		76.8 a	81.8 b	87.1 c	7.0 ^d	63.3 e	69.3 ^f	76.1 ^g	7.0 ^h	74.8 ⁱ	80.0 ^j	89.4 ^k	7.8 ¹

* Values are compared within columns.

*Values designated by the same alphabetical letter are not significantly different ($\alpha = 0.05$).

CONCLUSION

Single-pass drying of rough rice using high temperatures did not have an apparent effect on color, degree of milling, and thermal properties of pureline, long-grain, Wells, medium-grain, Jupiter, and hybrid, long-grain, CL XL729 rice cultivars. However, increasing temperatures and RHs had a significant effect on the pasting viscosity profile of rice flour from all three cultivars lots.

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VI. OVERALL CONCLUSIONS

Since high-temperature drying has been noted as one of the most important postharvest handling processes affecting rice quality, this dissertation was primarily focused on examining (1) the high-temperature drying characteristics of rough rice, (2) the effect of drying conditions on milling quality of rough rice, and (3) the effect of drying conditions on milled rice functionality. The overall goal of the dissertation was to develop a new method of drying rough rice by incorporating the glass transition principle, which would facilitate fast rate drying with minimal loss to milling quality and functional properties.

It was observed in the first experiment that rough rice could be dried at a very fast rate using high temperatures. The equilibrium moisture content of rough rice, which is dependent on the temperature and relative humidity of the drying air, could be maintained above or below glass transition, thereby controlling kernel material state during drying. The Page equation was successfully fitted to the drying data and was useful in predicting drying rate at the experimental conditions. The Modified Chung-Pfost equation was found adequate for predicting equilibrium moisture content of rough rice at high temperatures using empirical constants estimated from the experimental data. Future work requires that this drying study be extended to more rice cultivars, especially hybrids, which are quickly becoming the dominant seed in the rice industry. Incorporating more cultivars will make this work more practical to the rice industry by providing detailed descriptions of the drying characteristics of each cultivar currently being grown, thereby making the drying process more predictable and energy efficient.

In the second experiment, it was observed that controlling the material state of the kernel during drying coupled with proper tempering immediately after drying could eliminate the need to dry rough rice in multiple passes. The single-pass drying method has the potential of

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increasing dryer throughput, thus significantly reducing the long lines commonly observed at commercial drying facility during the harvest period. Future studies should focus on pilot scale testing of the drying method incorporating additional cultivars and higher drying air temperatures. In addition, the potential of applying this drying method to commercial cross-flow dryers should be investigated given that most rice in the United States is dried using cross-flow dryers.

The third experiment demonstrated that high drying air temperatures, when properly implemented, do not have deleterious effects on the functional properties of milled rice. Among the functional properties investigated, only pasting viscosity profile was affected by drying rough rice at high temperature drying. Color, degree of milling, and thermal properties were not affected. Future work should consequently focus on sensory analysis of cooked rice using trained panelists. In addition, aromatic rice cultivars such as Jasmine and Basmati should be incorporated in future studies to investigate the effect of this drying method on the volatile compounds. This study could also be extended to examine the relationship between the drying conditions and secondary products of milled rice such as baked foods, rice cereals etc,.