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Rice processing: milling and value-added effects

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RICE PROCESSING: MILLING AND VALUE-ADDED EFFECTS

A Thesis Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science in Biological and Agricultural Engineering

in

The Department of Biological and Agricultural Engineering

by Rebecca C. Schramm B.S., Louisiana State University and Agricultural and Mechanical College, 2004 August 2006

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ABSTRACT

The ultimate goal of this research is to characterize data from the laboratory, pilot, and industrial scale rice mills. Pilot and laboratory scale data are presented in this research. Two long grain rice cultivars were milled with two different scale mills. Cheniere and Cypress were milled with a McGill No. 2 mill and a pilot scale mill (Satake). Both material streams, rice kernels and bran, were collected and weighed. Measurements of Degree of milling, transparency, and whiteness were made with a milling meter (Satake). Yield and bran fraction were calculated. Samples of the bran were heat stabilized and prepared for high pressure liquid chromatography (HPLC). HPLC analysis determined the concentration of vitamin E and oryzanol. Parameter values were reported as laboratory, pilot, or category assignment of low, medium, and high. Yield values for both rice varieties and both mill scales were highest at the low category. Degree of milling measurements increased with increasing process time setting for the laboratory scale mill and with increasing operational mill setting for the pilot scale mill. DOM data divided by category showed an increase for both varieties and both mill scales from the low to high categories. Transparency and whiteness values increased from low to high category. At the laboratory scale mill, for Cheniere, the highest levels of vitamin E and oryzanol occurred at the 10 second mill setting. For Cypress, the highest level of vitamin E occurred at the 10 second mill setting, and the highest level of oryzanol resulted at the 5 second time setting. Category and pilot scale values for both vitamin E and oryzanol were highest at the low category or the lowest mill setting.

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CHAPTER 1 ─ INTRODUCTION

The world produced 619 million metric tons of rice in 2005 (United Nations, 2005), which accounts for nearly one fourth of the world's cereal grain crops. The United States produced between 11 and 12 million metric tons of rice, with Louisiana accounting for over one and half million metric tons of production in 2005 (USDA, 2006). In spite of this production level, Louisiana faces major problems as a result of two hurricanes, Katrina and Rita, which impacted agricultural production.

Louisiana's rice production was heavily impacted by Katrina's and Rita's devastation. Preliminary estimates of cumulative economic impact from these two hurricanes for the rice industry are over twelve million dollars. The state is expected to have agricultural impacts due to increased costs and reduced income of over one and a half billion dollars (LSU AgCenter, 2005).

Problems caused by the hurricanes include salt water intrusion in rice fields and the high energy cost of harvesting crops. A Louisiana State University AgCenter researcher, Johnny Saichuk, stated that the rice acreage in Vermillion parish will experience a decrease in acreage planted from 80,000 in 2005 to about 27,000 acres this year (Courreges, 2006). Other ricegrowing parishes are expected to experience decreases in acreage planted due to economic and physical field conditions.

A farmer in Vermillion parish reported that his fields had salt concentrations of 3000- 8000 parts per million (ppm); 750 ppm is the upper limit for rice production (Courreges, 2006). The Louisiana State University AgCenter extension agent for Vermillion parish expressed concern that salt damage and the recent drop in rice prices may cause rice farmers that let their fields lie fallow this year to not return to rice production (Courreges, 2006).

Rice milling involves several steps: removal of the husks or shell, milling the shelled rice to remove the bran layer, and an additional whitening step to meet market expectations for

appearance of the rice kernels. This process generates several streams of material which include the husks, the bran, and the milled rice kernels.

Studies have compared several different laboratory scale mills (Bautista & Siebenmorgen, 2002), examined the quality characteristics of rice produced from different types of commercial mills, and studied the effect of different size kernels on milling parameters (Rohrer & Siebenmorgan., 2004). As new varieties of rice are being introduced to increase yields, these new varieties of rice are tested at the laboratory scale for milling characteristics. Testing at the laboratory scale has not always predicted milling characteristics accurately at the industrial level. A recent example is with the rice variety, Cocodrie. Initial testing had shown a strong quality profile, but poor quality results occurred for late season harvest. A pilot scale study resulted in the determination of mill settings to optimize late season harvest milling quality (Hua, et al., 2006).

The pilot scale mill at Louisiana State University at Baton Rouge provided useful information regarding late season milling performance for this rice variety. From the success of this pilot scale project, questions arose about the predictability and reliability of data obtained at one scale as a basis for a different scale. This study addresses the laboratory and pilot scale mills with future work planned to extend this work to the industrial scale. A better understanding of the correlation between different scale mills would provide valuable information for both economic areas: the milling process itself and the value-added component of the rice industry.

Agricultural products result in multiple material streams when processed. By-products of traditional processing have often been under utilized (Perretti et al., 2003). More recent investigations have examined the composition of rice bran material and possible uses for this byproduct of the milling process. Studies have been conducted to determine the location of the most valuable components within the bran layer (Rohrer & Siebenmorgen, 2004). Milling times,

kernel-size and fraction (Siebenmorgen & Sun, 1993) variety and environmental conditions (Bergman & Xu, 2003) have been the focus of studies which examined factors influencing the concentration and location of components within the rice bran layer.

The objectives of this work were: (1) To correlate milling parameters between laboratory and pilot scale mills at given settings, (2) To quantify the amount of rice bran removed at selected settings, and (3) To characterize the amount of rice bran removed at a given setting with the concentration of vitamin E and oryzanol present in the rice bran for laboratory and pilot scales.

Figure 1.1, Laboratory Scale Mill: Model (M1) 2, H. T. McGill, Inc., Brookshire, Texas

Figure 1.2, Pilot Scale Mill: Model GPS300A, Satake Engineering, Co., Tokyo, Japan

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CHAPTER 2 –MILLING PARAMETER EFFECTS

Introduction

The world population exceeds 6.5 billion individuals and over half are dependent on rice for at least a portion of their diet (IRRI, 2006). Rice is an important grain crop to the world. For the rice industry, a high quality rice product at a profitable price is the goal. For the milling process, this translates to production of the highest quality whole rice kernel possible. As unbroken grains sell for higher prices in the market than broken kernels, yield is an important quality measurement. Whiteness, as reflected through degree of milling values, is a second very important industry measure of rice quality.

The effect of the milling process on the outcome of rice quality has been researched by examining the quality of the whitened rice product focusing on parameters such as degree of milling, transparency, whitening, and yield. In a study on milling characteristics for different kernel size fractions, the researchers examined different thickness fractions for several cultivars and found a linear relationship between head yield and degree of milling within each thickness fraction (Rohrer & Siebenmorgen, 2004). Bautista & Siebenmorgen (2002) compared several laboratory scale mills and determined that yields decrease with increasing milling duration. In the 10 to 50 second range for the McGill mill, they found that yield decreased in an approximately linear fashion with increasing process time. They noted that yields were affected by other factors including moisture content and variety (Bautista & Siebenmorgen, 2002).

Rice quality measurements have traditionally been made at the laboratory scale and utilized for predicting full scale mill performance. This processed failed with the long-grain rice variety, Cocodrie. Problems occurred with late season harvest at the full scale mill. The pilot scale mill (Satake Engineering, Co., Tokyo, Japan) at Louisiana State University was employed

to further study milling characteristics of Cocodrie. The optimization study provided useful information for the industrial scale (Hua et al., 2006). This example of the pilot scale mill's potential for better predicting milling characteristics of new rice varieties inspired questions about scaling effects for all three scale mills: laboratory, pilot, and industrial.

The eventual goal of this research is to characterize data for three scale mills: laboratory, pilot, and industrial. For this study, pilot and laboratory scale data are presented. The two scale mills used in this research vary greatly in operational characteristics and size. The laboratory scale mill operates as a batch process that is controlled by setting process times, and the pilot scale mill is a continuous process controlled by setting operational settings on the mill. To assist the comprehension of the size difference, the laboratory scale mill can be moved by one person and the pilot scale mill is approximately one sixth the size of a full industrial scale mill. The research data obtained can be presented by scale and by operational setting, but a method is needed to organize and compare data from such size diverse equipment. The industry supplied the solution to categorizing the data as rice millers tend to group information into categories of low, medium, and high categories. The categories of low, medium, and high were defined for both scale mills in order to facilitate valid comparisons.

For the pilot and laboratory scale, this study measured degree of milling, transparency, and whiteness; determined yield; and quantified bran removal. These parameters are then utilized to meet the study's objectives: (1) To correlate milling parameters between a laboratory scale mill and a pilot scale mill at given flow settings, and (2) To quantify the amount of rice bran removed at selected flow settings.

Materials and Methods

Two long grain rice varieties, Cheniere and Cypress, were shelled and milled utilizing a laboratory and a pilot scale mill. The amounts of shelled rice, milled rice, and bran material

removed were weighed for each replicate performed. Broken kernels of rice were removed from the milled rice and yield determined. Bran fraction was calculated, and degree of milling (DOM), transparency, and whiteness were measured.

Sample Preparation

Rough rice stock in sacks of 22.7 kilograms of two long grain rice varieties, Cypress and Cheniere, was supplied by the Louisiana State University AgCenter from the Crowley Rice Station and remained in cold storage (0°C) until required for experimentation. Rice was removed from cold storage the day before processing. This allowed time for the rice to equilibrate to room temperature. Before milling, the moisture content of each sack was measured. Rough rice moisture content was monitored using a grain analysis computer (Dickey-John, Model GAC II). Moisture content varied little between the rough rice materials sampled. The moisture content was measured between 14 and 14.5 percent for all of the rough rice milled (Appendix A).

Processing

Samples of the rough rice were shelled and milled, with samples of milled rice and stabilized bran collected from both scale mills. At the laboratory scale, separate shelling (McGill Sheller, Model MS1) and milling (McGill mill, Model (M1) 2, H. T. McGill, Brookshire, Texas) units were used in processing the rice samples. Samples of 175 grams were processed through the shelling unit to ensure a shelled sample size of 125 grams. A sample size of 125 grams was processed through the milling unit and the separate streams of milled rice and bran were collected and weighed. The shelled rice samples were processed in triplicate at nine time settings from five to 45 seconds in five second intervals, which is consistent with the range of time settings seen in the literature (Bautista & Siebenmorgen, 2002).

 The pilot scale mill (Satake Engineering, Co., Tokyo, Japan) operates in a series of unit operations. Rough rice enters the shelling unit (Model GPS300A, Satake Engineering, Co.,

Tokyo, Japan), and after shelling, is conveyed to the whitening unit (Model VAF10AM, Satake Engineering, Co., Tokyo, Japan) in a continuous operation. The pilot scale mill was divided into components for shelling and milling, creating separate unit operations that permitted the shelled rice samples to be weighed. Samples of 11.4 kilograms were shelled, and then samples of nine kilograms were measured and milled. A funnel was constructed and used to feed shelled rice to the whitening unit of the pilot scale mill (see Appendix A for details). Milled rice and bran were collected and weighed. Three replicates were made at each operational pilot scale mill settings. In practice, the pilot scale mill is run at operational settings of three, six, and nine, which are referred to as low, medium, and high settings (Hua et al., 2006).

Statistical Analysis

Table 2.1 and Table 2.2 present the experimental design. The experiment was performed in triplicate (as indicated by the small red three in Tables 2.1 and 2.2) with random selection of process time settings for the laboratory scale mill and random selection of operational pilot scale mill settings. Fifty-four replicates for two varieties and nine time settings were performed at the laboratory scale, and eighteen replicates were performed at the pilot scale for two varieties and three operational mill settings. Microsoft Excel Data Analysis Tools were utilized to analysis data. Specifically, a student-t test with two samples assuming equal variance was employed in statistical calculations with a test alpha of 0.05. Materials were presented in graphical form utilizing Microsoft Excel graphical package.

The experiment's design was approved by the experimental statistics department at Louisiana State University. The experiment collected data for two rice varieties at two mill scales, laboratory and pilot. Measurements were made to determine the amount of bran removed and the percent of unbroken kernels. For each replicate, degree of milling, transparency, and whiteness were measured for a sample of milled rice. Data tables are presented in Appendix B.

Run	Time (Seconds)	Shelled Rice (grams)	\sim Milled Rice (grams)	Unbroken (grams)	Milled Rice DOM, T, W	Bran (grams)	Stabilized Bran (grams)
	5						
	10						
	15						
	20						
	25						
	30						
	35						
	40						
	45						

Table 2.1, Laboratory Experimental Design

Table 2.2, Pilot Experimental Design

Measurements

Milled rice samples were weighed, weight recorded, and processed with a rice shaker table (Shaker Table, Model 61-115-60, Grainman Machinery Co., Miami, Florida) to remove broken kernels of rice. Different size trays were used in the shaker table to remove the broken kernels. Sorter trays of size 10 and size 12, used at the top and bottom positions of the shaker table respectively, removed the broken kernels from the milled rice sample. Degree of Milling (DOM), transparency, and whiteness readings were taken using a Satake Milling Meter (Satake Engineering Co., Tokyo, Japan). Three replicate measures of each sample were taken, stored in the meter, and the mean of these values, which was determined by the meter, recorded as the degree of milling value for each sample.

Degree of milling, transparency, and whiteness are important quality standard for the rice industry. The Satake milling meter (Model MM1-B, Tokyo, Japan) provides a quick and accurate method for measuring transparency and whiteness, and determining DOM, a measure of the

amount of the bran layer and germ removed by processing. This meter uses an optical method, employing the properties of light, both refraction and transmission to provide measures of whiteness, transparency, and an internal algorithm to calculate degree of milling (Satake Rice Milling Meter MM1C Specification Sheet, 2005). There have been several methods to determine degree of milling, however, currently the Satake milling meter is widely used. Other methods require experienced personnel or lengthy test procedures (Deobald & Hogan, 1961). The Federal Grain Inspection Service uses a visual classification method of four categories to classify milled rice samples into under milled, lightly milled, reasonably well milled, or well milled divisions. Classification by this method requires experienced personnel. The petroleum-ether extraction test, an industry standard for many years, is a very lengthy procedural test (Siebenmorgen & Sun, 1994). The Satake milling meter employs light properties to provide a rapid measurement of degree of milling, reporting values on a scale of 0 to 199.

An industry standard for rice quality evaluation, yield or whole kernel yield is determined by dividing the weight of unbroken milled rice kernels by the amount of shelled rice processed (Bautista & Siebenmorgen, 2002). The USDA standard defines an unbroken kernel as at least $\frac{3}{4}$ of a kernel for yield determination (Siebenmorgen $\&$ Sun, 1994). For yield calculations, the weight of the shelled rice that is to be milled needs to be recorded for both scale mills. The shelled rice weight for the laboratory scale mill, which is a batch process, was easily measured. However, the pilot scale mill is a continuous process; and to permit the weight of the shelled rice to be measured before milling, the pilot scale mill procedures were conducted as unit operations. The rough rice was shelled, the shelled rice weighed, and the shelled rice was then milled.

The amount of bran removed from shelled rice is a measure of the quality of the milled rice. Bran removal is calculated as the amount of bran removed divided by the weight of the shelled rice processed. The amount of bran can be directly measured or calculated as the

difference in the weight of shelled rice milled and the weight of milled rice. This value is often reported as a percentage. To achieve a well-milled sample, the Standards Committee of the American Association of Cereal Chemists in 2000 suggests a goal of a 0.12 or twelve percent reduction in the difference between shelled rice and milled rice weights during the milling process (Bautista & Siebenmorgen, 2002).

Results and Discussion

Results

Results are presented in three subsections to facilitate data interpretation: (1) yield, (2) quality measurements of Degree of Milling (DOM), transparency, and whiteness, and (3) mass of bran removed.

Yield

Figures 2.1 and Figure 2.2 present laboratory and pilot scale yield data by scale for both Cypress and Cheniere. P-values (α =0.05) indicate that there is no statistical difference between the two varieties of rice, Cheniere and Cypress, for yield determination at the laboratory scale. From the statistical comparison of the pilot scale yield for Cypress and Cheniere, p-values ranged from just over 0.05 to 0.9 (α =0.05), indicating that no statistical difference exists. There is a statistical difference between values at setting 3 and setting 9 within a variety at the pilot scale.

Bautista and Siebenmorgen (2002) studied two rice varieties with a McGill Mill at 10, 30, and 45 second milling times. In their study the long grain variety, Drew, had yield percentages ranging from 60 to 70 percent. In this study at the laboratory scale, the yield percentages range from 79.3 to 83.7 for the Cypress variety and from 78.0 to 89.0 for the Cheniere variety. The pilot scale values are from 66.9 to 86.9 for Cypress and from 73.8 to 86.8 for Cheniere. For both

the laboratory and pilot scale mills, there is a decrease in yield with an increase in process time setting or operational mill setting. Data tables are presented for yield values in Appendix B.

Figure 2.1, Yield values laboratory scale mill

Figure 2.2, Yield values pilot scale mill

In the variety comparisons, the lowest process time setting for the laboratory scale mill and the lowest operational mill setting for the pilot scale mill resulted in the highest yields for the respective scale mill. The low time setting of 5 seconds and the low mill setting exhibit no statistical difference when compared for both rice varieties (Cheniere p-value = 0.052, Cypress p-value = 0.318). Table 2.3 contains the values for the 5 second laboratory mill process time

setting and the low (3) pilot scale operational mill setting. This is a possible point for scaling between the laboratory and pilot scale mills for yield as these points exhibited no statistical difference.

		Cheniere	Cypress					
Low Setting For Mill	Value	Standard Deviation	Value	Standard Deviation				
Laboratory (5 seconds)	88.69	0.49	83.67	4.71				
Pilot (Setting 3)	86.91	1.22	86.91	0.88				

Table 2.3, Yield in Percent for Lowest Setting Tested for Mills Yield (Percent)

Quality Measurements

Quality measurements are degree of milling (DOM), transparency and whiteness. The results of each measurement are presented and discussed. Data tables are located in Appendix B.

Degree of Milling

Figures 2.3 and Figure 2.4 present laboratory and pilot scale data, respectively. The laboratory scale mill data had a range of values for degree of milling from 40 to almost 125. There is no statistical difference in mean degree of milling readings between rice varieties at measured time settings for the laboratory scale mill (P-values: 0.06 to $0.90 > \alpha = 0.05$). For the Cypress variety at the laboratory scale, there is no statistical difference between successive values until after the 20 second setting. Cheniere values are not statistically different between successive values until after the 30 second setting. Pilot scale degree of milling readings are characterized by a narrow range of values from the low nineties to a few points over one hundred, with no statistical difference (P-values: 0.35 to $0.69 > \alpha = 0.05$) indicated between varieties.

Figure 2.3, Degree of Milling Laboratory Scale

Figure 2.4, Degree of Milling Pilot Scale

The graphical representations (Figure 2.5 and Figure 2.6) of the comparisons of the different scale mills for DOM readings illustrate a general trend of increasing DOM readings with increasing flow rate setting at the pilot scale and increasing process time at the laboratory scale. The laboratory scale time settings, reported on the left hand vertical axis, indicate increasing process times from 0 to 45 seconds. The pilot scale values are reported as operational mill settings and represent an increase in flow rate with increasing operational setting. In Figure 2.5 and Figure 2.6, there are points of interest where the DOM values are similar for both scale mills. These points occur at the lowest mill setting of 3 and both the 15 and 20 second process time settings for the laboratory mill. P-values indicate that there is no statistical difference for Cypress or Cheniere for pilot scale DOM values at a setting of 3 and for both the 15 and 20 second time settings of the laboratory mill. Table 2.4 presents the actual degree of milling values for these settings.

 Figure 2.5, Degree of milling comparison both scale mills

 Figure 2.6, Degree of milling comparison both scale mills

	Rice Variety					
Mill (Setting)	Cheniere	Cypress				
Pilot (3)	99.33	93.33				
Laboratory $(15$ seconds)	94.33	96.0				
Laboratory $(20$ seconds)	101.67	106.33				

Table 2.4, Comparison of DOM values

Siebenmorgen and Sun (1994) recorded degree of milling values for three cultivars.

Table 2.5 contains their data for 15, 30, and 45 second settings for a McGill mill in parallel with the data obtained in this work at the same process time settings. DOM increased from 15 to 30 second settings much faster than the increase from the 30 to 45 second setting for both Cheniere and Cypress. This pattern is consistent with the values form Siebenmorgen and Sun contained in Table 2.5.

Table 2.0, Decree of Mining Paraco								
Mill Time		Sun and Siebemoregen Data	Current Study Data					
Settings	New Bonnet Mille		Lemont	Cheniere	Cypress			
			59	94				
			88	-16				

Table 2.5, Degree of Milling Values

Transparency and Whiteness

Transparency, whiteness, and DOM values are presented by mill scale in Figures 2.7 and 2.8. Figure 2.7 presents the data for DOM, transparency, and whiteness measurements made at the laboratory scale for both varieties of rice. It is interesting to note that the DOM and transparency curves for the laboratory scale when plotted against process time settings have a

very similar shape. Whiteness values present very different shapes by variety. Pilot scale values for whiteness are almost indistinguishable, while transparency values present similarly shaped curves.

Bergman and Xu (2003) measured whiteness as a part of a study on genotype and environmental effects on tocopherol, tocotrienol, and oryzanol concentrations. In a table of trait levels across cultivars, years, and growing locations, a mean whiteness level was recorded as 41.84. Whiteness values presented in Figures 2.7 and 2.8 are consisted with this study for the laboratory and pilot scale mills, and for the Cheniere and Cypress varieties.

Figure 2.7, Laboratory Scale Quality Measurements

 Figure 2.8, Pilot Scale Milling Meter Measurements

Bran

Bran removed as a function of process time setting and operational mill setting are reported as fractions of the total bran removed in Figure 2.9 and Figure 2.10. At the laboratory scale, the bran fraction (Figure 2.9) removed increases with process milling time. Statistically there is no difference between the varieties for the 5 and 10 second settings, beyond the 10 second setting most settings exhibit a statistically difference.

Figure 2.9, Bran fraction laboratory scale

Pilot scale bran fraction has a very narrow range of values from 0.10 to 0.12. Statistically (p-values $\lt \alpha$ = 0.05), the bran fractions at the pilot scale are different between varieties (Figure 2.10). Data table on bran fraction are presented in Appendix B.

Velupillai and Pandey (1987) reported that 65 to 73 percent of the bran can be expected to be removed at the 20 second laboratory mill setting. Data from this study shows an 86 percent level of bran removal for the Cypress variety and an 82 percent level of bran removal for the Cheniere variety. The comparison gives an idea that the values are reasonable.

In a study using a McGill mill, Model Number 2, Watson et al. (1975) found that at the 30 second time setting for a reasonably well-milled to well-milled sample, a percent weight loss of seven to eight is expected. In this study and using the 40 second time setting as basis, the Cypress variety at the 30 second time setting has a percent of bran removal of 92 percent, or a weight reduction of eight percent. A seven percent reduction in weight occurs for the Cheniere variety with a 93 percent level of bran removal.

The Standards Committee of the American Association of Cereal Chemists recommended a twelve percent reduction in weight as the goal for milling. The target value of twelve percent

weight reduction is expected to occur at the 30 second time setting for the laboratory mill. In one study which included rice varieties, Drew and Bengal, the twelve percent reduction for rice varieties was obtained at the 30 second time settings on the laboratory scale (McGill, Model 2) (Bautista & Siebenmorgen, 2002). For this study at the 30 second time setting on the laboratory scale mill, the bran fraction was calculated to be 0.124 for the Cypress variety; and for the Cheniere variety, the bran fraction was 0.120. Both of these values meet the target of a twelve percent reduction in weight between shelled and milled rice samples during processing. At the pilot scale for the Cypress variety, the mill setting of 6 produces a bran fraction of 0.121. At the pilot scale mill setting of 9, the Cheniere variety has a reduction in weight that results in a bran fraction of 0.116.

There is a general trend of increasing bran fraction with increasing process time setting (Figure 2.9). Pilot scale bran removal values exhibit little change across mill settings for both varieties (Figure 2.10). There is a point of interest where both varieties of rice have similar bran fractions for both scale mills (Figure 2.11 and Figure 2.12). This point occurs at the 15 second laboratory process time setting and the pilot scale operational mill setting of 3. Statistical analysis reveals no statistical difference for this point of interest for both varieties of rice (Cheniere: p-value =0.18, α =0.05 and Cypress: p-value=0.44, α =0.05).

Scaling Effects

The ultimate goal of this research is to correlate data from the laboratory, pilot, and industrial scale mills. Pilot and laboratory scale data are presented in this research. Rice millers tend to group information into categories of low, medium, and high categories. The categories of low, medium, and high were defined for both scale mills in order to facilitate valid comparisons.

 Figure 2.11, Bran fraction comparison different scale mills

 Figure 2.12, Bran fraction comparison different scale mills

The pilot scale data is easily assigned to a category as there are only three settings to accommodate. The low category is the value from the setting 3, the medium value is the setting 6, and the high value is assigned as pilot mill setting 9. After pilot scale category assignment, laboratory scale data can be grouped and categorized.

As the laboratory scale mill is a batch process, there are no flow rates but times of processing. There are nine processing time settings for the laboratory scale mill. One approach is to divide the number of pilot scale settings into the number of laboratory scale process time

settings. This results in assigning three process time settings to each pilot scale mill setting. For the pilot scale, there are three flow rates, allowing the smallest value of the flow rates tested to be assigned as low, the middle level flow rate to be assigned as medium, and the fastest flow rate to be assigned as high. The laboratory scale categories are assigned by process time settings. The lowest three time settings of 5, 10, and 15 seconds are assigned as the low category, the 20, 25, and 30 second tine settings are assigned as medium, and the 35, 40 and 45 second time settings are assigned as high. These divisions are expected to create directly comparable categories of low for the laboratory scale mill with the low category of the pilot scale mill. Trends and comparisons are presented in conclusions. Table 2.6 presents the data for the laboratory scale and pilot scale in flow rate categories of low, medium, and high for all parameters considered. Figures 2.13 to 2.18 present categorized data for yield, for degree of milling, and for bran fraction.

Summary: Values at Flow Rate Categories									
Rice Variety:			Cheniere		Cypress				
	Category	Laboratory Scale		Pilot Scale		Laboratory Scale		Pilot Scale	
Parameter		Value	Standard Deviation	Value	Standard Deviation	Value	Standard Deviation	Value	Standard Deviation
	Low	87.29	1.50	86.91	1.22	82.46	1.07	86.83	0.88
Yield (Percent)	Medium	83.40	2.32	76.93	1.92	82.20	1.45	79.33	1.52
	High	80.00	1.82	66.91	3.87	80.41	1.19	73.81	2.14
	Low	72.11	24.20	99.33	1.15	74.89	26.13	93.33	9.87
Degree of Milling	Medium	108.89	7.17	102.00	3.00	109.44	3.17	103.00	2.65
	High	122.11	4.99	106.33	3.06	123.89	3.56	108.00	3.00
	Low	3.03	0.63	3.93	0.27	3.12	0.49	4.07	0.27
Transparency	Medium	3.45	0.36	4.05	0.13	3.46	0.10	4.22	0.20
	High	3.65	0.02	4.06	0.25	3.59	0.16	4.22	0.26
	Low	34.92	5.07	39.07	0.32	34.92	5.07	37.53	1.78
Whiteness	Medium	38.88	2.43	39.47	0.86	38.88	2.43	39.5	0.85
	High	40.62	1.30	40.37	1.04	40.62	1.30	40.5	1.00
	Low	0.07	0.03	0.11	0.02	0.07	0.04	0.11	0.01
Bran Fraction	Medium	0.11	0.01	0.11	0.01	0.12	0.00	0.12	0.01
	High	0.13	0.01	0.12	0.00	0.14	0.00	0.13	0.00

Table 2.6, Summary Parameters in Flow Rate Categories

Figure 2.13, Yield by Category

Figure 2.14, Yield by Mill Scale

Figure 2.15, DOM by Category

Figure 2.16, DOM by Mill Scale

Figure 2.17, Bran Fraction by Category

Figure 2.18, Bran Fraction by Mill Scale

Conclusions and Recommendations

Data obtained in the study was presented by scale, by variety, and by category. The conclusions and recommendations are presented with this format. The results by category characterize scaling between the laboratory and pilot scale mills.

Laboratory values for yield showed little change across all time settings for both varieties of rice tested. Pilot scale yield values decreased with increasing operational mill setting for both Cheniere and Cypress varieties. Yield values for both rice varieties and both mill scales were highest at the low category.

Degree of milling measurements increased with increasing process time setting for the laboratory scale mill and with increasing operational mill setting for the pilot scale mill. Data divided by flow rate category showed an increase for both varieties and both mill scales from the low to high categories. At the medium category, laboratory scale mill values predicted the pilot scale values within several points. Transparency and whiteness values followed similar patterns of increase from low to high category as DOM measurements displayed.

The amount of bran removed at the laboratory scale increased with process time setting. Clear divisions of bran fraction were observed at the laboratory scale by time setting. At the pilot scale, the amount of bran removed showed little variation between operational mill settings. Laboratory scale mill data presented within categories showed distinct and increasing bran removal from the low to the high category. Categorization of pilot scale data created separate divisions for each category for the Cypress variety. The Cheniere variety data showed the same value for low and medium categories.

This work should be expanded to the industrial scale. Additional varieties should be tested for all scale mills. Further study of bran fraction at the pilot scale to determine the reason for the difference observed between the two varieties tested.

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CHAPTER 3 ─ VALUE-ADDED EFFECTS FOR COMPONENTS IN THE RICE BRAN LAYER

Introduction

Rice is currently important to the diet of a quarter of the world's population (IRRI, 2006). The rice milling process produces several steams of material, including husks, milled rice, and bran. Rice bran is a by-product of the milling process. Often by-products are under utilized; the area of value-added processing has become increasingly important as a way to increase economic rates of return. Rice bran is widely used as a feedstock for animals (Perretti et al, 2003). However, numerous compounds with important health effects for humans have been identified in rice bran. Milling times, kernel-size and fraction (Siebenmorgen & Sun, 1993), variety and environmental conditions (Bergman & Xu, 2003) have been the focus of studies which examined factors influencing the concentration and location of components within the rice bran layer. Duvernay et al. (2005) have conducted a study on the microwave extraction of antioxidant components from rice bran.

Rice bran contains antioxidants, anti-tumor compounds and possibly other constituents with health benefits (Qureshi et al., 2000). Various epidemiological studies have shown that antioxidants reduce oxidative damage to bimolecular structures, which plays a role in prevention of chronic diseases. Antioxidants may help retard the onset of diabetes and Alzheimer's disease, and may help prevent heart disease and cancer (Adom & Liu, 2002). In a study on rice bran oil, two groups of compounds found in the unsaponifiable portion of the rice bran oil were identified as tocotrienols and gamma-oryzanol (Rogers et al., 1993). Tocotrienols, which are members of the vitamin E family, and gamma-oryzanol, are being studied for their potential health benefits (Rodgers et al., 1993). These compounds have been identified as having antioxidant activity that are of interest to the pharmaceutical and nutraceutical industries.

The eventual goal of this research is to characterize data for three scale mills: laboratory, pilot, and industrial. For this study, pilot and laboratory scale data are presented. The two scale mills used in this research vary greatly in operational characteristics and size. The laboratory scale mill operates as a batch process that is controlled by setting process times, and the pilot scale mill is a continuous process controlled by setting operational settings on the mill. To assist the comprehension of the size difference, the laboratory scale mill can be moved by one person and the pilot scale mill is approximately one sixth the size of a full industrial scale mill. The research data obtained can be presented by scale and by operational setting, but a method is needed to organize and compare data from such size diverse equipment. The industry supplied the solution to categorizing the data as rice millers tend to group information into categories of low, medium, and high categories. The categories of low, medium, and high were defined for both scale mills in order to facilitate valid comparisons.

Although much work has been done to characterize high-value components of rice bran, there is a dearth of literature on scale-up of this process for use at the industrial scale. This study seeks to quantify the location of vitamin E and oryzanol in the rice bran layer in the context of the influence of scale on this process. The objectives of this study are: (1) To quantify the amount of rice bran removed at selected flow settings, and (2) To correlate the amount of rice bran removed at a given flow setting with the concentration of antioxidant present.

Materials and Methods

Two varieties of rice, Cheniere and Cypress, were processed using a laboratory and a pilot scale mill. Data was collected for scaling studies of milling parameters (See details in Chapter 2.) and bran research. This chapter examines the amount of bran removed at given mill settings and concentration of several antioxidant components present in the bran layer. For each replicate, five gram bran samples were collected and heat stabilized (see Appendix A for details)

for high pressure liquid chromatography (HPLC) determination of vitamin E and oryzanol concentration. Scale effects are examined between the laboratory and pilot scale mills.

Sample Preparation

Two varieties of long grain rice, Cheniere and Cypress, were supplied as sacks of rough rice (approximately 23 kilograms) by the Louisiana State University AgCenter from the Crowley Rice Station and remained in cold storage (0°C) until required. The day before processing, rice was removed from cold storage. This allowed time for the rice to equilibrate to room temperature. Before milling, the moisture content of each sack was measured with little variance noted between samples (refer to Appendix A for moisture content data).

Processing

Samples of the rough rice were shelled and milled, with samples of milled rice and stabilized bran collected from both scale mills. At the laboratory scale, separate shelling (McGill Sheller, Model MS1) and milling (McGill mill, Model (M1) 2, H. T. McGill, Brookshire, Texas) units were used to process the rice samples. Samples of 175 grams were processed through the shelling unit to ensure a shelled sample size of 125 grams. A sample size of 125 grams was processed through the milling unit and the separate streams of milled rice and bran were collected and weighed. The shelled rice samples were processed in triplicate at nine time settings from five to 45 seconds in five second intervals, which is consistent with the range of time settings seen in the literature (Bautista & Siebenmorgen, 2002).

 The pilot scale mill (Satake Engineering, Co., Tokyo, Japan) operates in a series of unit operations. Rough rice enters the shelling unit (Model GPS300A, Satake Engineering, Co., Tokyo, Japan), and after shelling, is conveyed to the whitening unit (Model VAF10AM, Satake Engineering, Co., Tokyo, Japan) in a continuous operation. The pilot scale mill was divided into components for shelling and milling, creating separate unit operations that permitted the shelled

rice samples to be weighed. Samples of 11.4 kilograms were shelled, and then samples of nine kilograms were measured and milled. A funnel was constructed and used to feed shelled rice to the whitening unit of the pilot scale mill (see Appendix A for details). Milled rice and bran were collected and weighed. Three replicates were made at each operational pilot scale mill settings. In practice, the pilot scale mill is run at operational settings of three, six, and nine, which are referred to as low, medium, and high settings (Hua et al., 2006).

The pilot mill equipment and the industrial mill equipment operate as a continuous process until the feed stream of rice is stopped. As a result for the pilot scale mill, flow rates were determined and correlated to operational mill setting in anticipation that this study being extended to include the industrial scale (see Appendix B for flow rates).

Five gram bran samples were collected at the laboratory and pilot scale for each replicate. This resulted in fifty-four bran samples at the laboratory scale, and eighteen samples at the pilot scale. These samples were heat stabilized (see Appendix A for details) and stored at minus eighteen degrees Celsius.

Statistical Analysis

Table 3.1 and Table 3.2 present the experimental design. The experiment was performed in triplicate (as indicated by the small red three in Tables 3.1 and 3.2) with random selection of laboratory scale mill process time settings and random selection of operational pilot scale mill settings. Fifty-four replicates for two varieties and nine time settings were performed at the laboratory scale, and eighteen replicates were performed at the pilot scale for two varieties and three operational mill settings. Microsoft Excel Data Analysis Tools were utilized to analysis data. Specifically, a student-t test with two samples assuming equal variance was employed in statistical calculations with a test alpha of 0.05. Materials were presented in several formats utilizing the graphical tools in Microsoft Excel. Data tables located in Appendix B.
Run	Time (Seconds)	Shelled Rice (grams)	Milled Rice (grams)	$\overline{}$ Unbroken (grams)	Milled Rice DOM, T, W	Bran (grams)	Stabilized Bran (grams)
	5	3	3	3	3	3	3
	10	3	3	3	3	3	3
	15	3	3	3	3	3	3
	20	3	3	3	3	3	3
	25	3	3	3	3	3	3
	30	3	3	3	3	3	3
	35	3	3	3	3	3	3
	40	3	3	3	3	3	3
	45	3	3	3	3	3	3

Table 3.1, Laboratory Experimental Design

Table 3.2, Pilot Experimental Design

Run	Mill Setting	Flow Rate (grams/sec)	Shelled Rice Milled Rice Unbroken (grams)	(grams)	(arams)	Bran (grams)	Stabilized Bran (grams)

The experiment's design was approved by the experimental statistics department at Louisiana State University. The experiment collected data for two rice varieties at two mill scales, laboratory and pilot. Measurements were made to determine the amount of bran removed and the percent of unbroken kernels. For each replicate, degree of milling, transparency, and whiteness were measured for a sample of milled rice. Samples of rice bran were collected for high pressure liquid chromatography (HPLC) to determine vitamin E and oryzanol concentrations.

Measurements

Table 3.3 presents a sample (one replicate of nine time settings for the laboratory scale mill) of the data obtained from HPLC tests performed to determine the concentration of vitamin E and oryzanol present in micrograms per gram of rice bran (see Appendix C for additional data and details). For vitamin E detection, a fluorescence detector with excitation wavelength of

290nm and an emission wavelength of 330nm was employed. A LC─Si Sulpecosil column (25 centimeter long 4.6 centimeters in diameter with a particle size of 5 micrometers) was used. The mobile phase consisted of hexane, acetic acid and ethyl acetate (99:0.5:0.5). The flow rate for the mobile phase for vitamin E detection was 2.2 milliliters per minute. For oryzanol, absorbance detection at a wavelength of 330 nm was used. HPLC was conducted with a C18 column and a mobile phase of methanol, acetonitrile, dichloromethane, and acetic acid (50:44:3:3) at a flow rate of 1.6 milliliters per minute.

							Table 3.3, Table of Data (Formatted by the technical specialist who conducted the tests		
	Vitamin E - Rice Bran						uq/q	ug/g	
ug	a-T	а-Т3	$r-T$	$r-T3$	ory	wt.(g)	vitamin_E	Orv	
a11	36.8	26.9	8.3	27.9	1124	0.5036	198.31	2231.28	
a12	34.5	32.1	6.3	34.7	1056	0.4991	215.57	2114.91	
a13	38.8	32.3	6.7	32.7	1188	0.4953	222.99	2398.84	
a14	37.2	32.7	7.2	37.6	1177	0.5053	227.07	2329.13	
a15	31.4	30.7	6.5	33.5	1018	0.4852	210.25	2098.25	
a16	30.1	25.6	6.1	29.9	867	0.4985	183.76	1740.00	
a17	25.3	23.7	5.4	32.4	1014	0.5090	170.73	1993.12	
a18	25.5	23.8	6.4	29.5	841	0.4895	174.04	1718.06	
a19	31.8	30.0	6.7	34.1	1222	0.4948	207.51	2469.86	

Table 3.3, Table of Data (Formatted by the technical specialist who conducted the tests)

Results and Discussion

Results

Results are presented in three subsections to facilitate data interpretation: (1) vitamin E,

(2) oryzanol, and (3) bran.

Vitamin E

The concentration of vitamin E for Cheniere and Cypress at the laboratory scale is shown in Figure 3.1. The largest amount of vitamin E was present at the 10 second setting for the laboratory mill for both Cypress and Cheniere varieties of rice. At the 10 second setting, Cypress contained 218 micrograms of vitamin E per gram of rice bran milled, and Cheniere contained

230 micrograms of vitamin E. Statistically at the 10 second process times, there is no difference in the amount of vitamin E contained in bran from either variety of rice (p-values $> \alpha = 0.05$).

 Figure 3.1, Vitamin E values for laboratory scale mill

Pilot scale vitamin E data is presented in Figure 3.2. The highest concentration of vitamin E is at the lowest tested operation mill setting, 3. Cypress had 198 micrograms of vitamin E per gram of rice bran milled while Cheniere contained 210 micrograms of vitamin E. Statistically the varieties exhibited no different (p-values: range, α =0.05) when compared at each mill setting. Both varieties exhibited a decrease in vitamin E concentration with increasing mill setting.

Figure 3.2, Vitamin E values for the pilot scale mill

The amount of vitamin E at the 15 second process time setting for the laboratory scale mill and low (3) mill setting for the pilot scale mill are close in terms of micrograms of vitamin E per gram of rice bran. For the Cypress variety at the 15 second setting, the concentration of vitamin E is 195 micrograms per gram of rice bran; and for the setting 3 at the pilot scale, the concentration of vitamin E is 198 micrograms per gram of rice bran. The data for the Cheniere variety is 215 micrograms of vitamin E per gram of rice bran at the laboratory scale mill setting of 15 seconds; and at the pilot scale mill setting of 3, the concentration of vitamin E is 210 micrograms. For both varieties of rice, there is no difference statistically between the 15 second laboratory scale value and the pilot scale value at setting 3 (Cheniere p-value = 0.84, Cypress pvalue = 0.93 , $> \alpha = 0.05$).

Oryzanol

For Cheniere and Cypress, the laboratory scale values for oryzanol are reported in Figure 3.3, and pilot scale values are reported in Figure 3.4. (Note: The 30 second laboratory scale value for Cheniere contains one less data point. See Appendix D for details.) There was a difference between varieties in terms of the bran fraction with the highest oryzanol concentration. For Cypress, the highest oryzanol concentrations occurred at the 5 and 15 second process time settings with 2516 and 2481 micrograms per gram of rice bran, respectively. The highest concentrations of oryzanol for Cheniere occurred at the 5 and 10 second process time settings with 2671 and 2699 micrograms per gram, respectively. At the pilot scale, there was no statistical difference between the varieties at the tested settings and a general downward trend in oryzanol concentration with increasing mill setting was observed. The highest concentrations of oryzanol occurred at the lowest operational pilot mill setting of 3, where Cypress had 2815 micrograms per gram of rice bran and Cheniere had 2388 micrograms per gram.

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In a study on nutraceutical concentrations within the bran layer, Rohrer and Siebenmorgen (2004) determined that the highest concentration of oryzanol occurred at the 10 second milling time for a McGill Mill No. 2. Cypress had the highest oryzanol content at the 5 second setting with 2516 micrograms per gram of bran; Cheniere had the highest level of oryzanol at the 10 second setting with 2699 micrograms per gram.

 Figure 3.3, Oryzanol values for laboratory scale mill

 Figure 3.4, Oryzanol values for the pilot scale mill

Bergman and Xu (2003) in a study of genotypes and environmental influence on vitamin E and oryzanol concentrations found a mean vitamin E value of 269.3 milligrams per kilogram and for oryzanol a mean value of 4263.56 kilograms per kilogram (or micrograms per gram) of rice bran. For this study at the 30 second setting, Cypress contains 187.58 micrograms per gram of vitamin E and 2161.72 micrograms per gram of oryzanol; and Cheniere contains 208.29 micrograms per gram of vitamin E and oryzanol 2114.77 micrograms per gram. This study's values are lower than the literature possibly due to extraction differences (saponification).

Bran

Bran removed as a function of process time setting and operational mill setting are reported as fractions of the total bran removed in Figure 3.5 and Figure 3.6. At the laboratory scale, there was a general trend of increasing bran fraction with increasing process time setting.

The pilot scale bran fraction data exhibited a narrow range of values from 0.10 to 0.12 and a statistical difference between the varieties at all mill settings tested (p-values $\leq \alpha = 0.05$). The lower laboratory mill process time settings and the lower pilot scale mill settings contain the smallest amount of bran and the highest levels of vitamin E and oryzanol.

Scale Effects

As stated in Chapter 2, the eventual goal of this research is to correlate data from the laboratory, pilot, and industrial scale mills. For this study, data for the laboratory and pilot scale mills was obtained and grouped into low, medium, and high categories. As the pilot scale mill was operated at three mill settings of 3, 6, and 9, categories were assigned as low for setting 3, medium for setting 6, and high for setting 9. After pilot scale category assignment, laboratory scale data can be grouped and categorized. The laboratory scale mill was operated at nine process time settings. The lowest three time settings of 5, 10, and 15 seconds were assigned as

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Figure 3.5, Laboratory Scale Bran Fraction

Figure 3.6, Pilot Scale Bran Fraction

the low category, the 20, 25, and 30 second time settings were assigned as medium, and the 35, 40 and 45 second time settings were assigned as high. The values of bran fraction, vitamin E, and oryzanol for each category are displayed in Table 3.4 and Figures 3.7 to 3.12. The percent change between categories is presented in Table 3.5.

Conclusions and Recommendations

Data obtained in the study was presented by scale, by variety, and by category. The conclusions and recommendations are presented with this format. The results by category characterize scaling between the laboratory and pilot scale mills.

The highest levels of vitamin E measured occurred at the 10 second setting for the laboratory mill and at the 3 setting for the pilot scale mill for both varieties of rice tested. For Cheniere and Cypress, the low category exhibited the highest level of vitamin E, with the values trending down in value from the low to the high category.

For oryzanol at the laboratory scale mill, the highest values occurred at the 10 second setting for Cheniere, and at the 5 second setting for Cypress. The highest oryzanol values at the pilot scale occurred at setting 3 for both Cypress and Cheniere. The low category for both varieties of rice contained the highest level of oryzanol.

The amount of bran material removed increased with increasing process time setting at the laboratory scale. At the pilot scale, bran removal occurred over a very narrow range of bran fraction, indicating little change between mill settings. The antioxidant compounds studied were found to be in the outer portion of the bran layer as the highest concentrations resulted from the shortest process times or lowest mill setting. This provided an additional potential for adding value as the highest levels for the two antioxidants examined occur at the lower time settings for the laboratory mill or the lowest mill setting for the pilot scale mill. The lower settings for both scale mills have the higher or highest levels of antioxidant contained in a smaller amount of bran

Table 3.4, Values of bran fraction, vitamin E, and oryzanol at specified categories

Table 3.5, Percent change between categories for parameter values of bran fraction, vitamin E, and oryzanol for both the Cypress and Cheniere varieties of rice

a refer to recommendations

d refers to a decrease between the first value and second value in comparison

Figure 3.7, Vitamin E by Category

Figure 3.8, Vitamin E by Mill Scale

Figure 3.9, Oryzanol by Category

Figure 3.10, Oryzanol by Mill Scale

Figure 3.11, Bran Fraction by Category

Figure 3.12, Bran Fraction by Mill Scale

 by weight. To extract the antioxidants of interest, a smaller amount (by weight) of rice bran would need processing. Between the low to medium categories for the laboratory scale, for Cheniere, bran material increased by 57 percent, and for Cypress, the increase was 43 percent. Operating at settings within the low category would substantially reduce the quantity of bran requiring processing. This represents a reduction in costs for processing, and handling, or transportation of the bran material which is to be used as a raw material for processing.

This work should be extended to the industrial scale. Additional studies should be conducted at the low end of the pilot scale mill's operational mill setting scale to identify if higher antioxidant concentrations are obtained. Examine the reason or reasons for the narrow range of bran fraction removed at the pilot scale, examining factors including: retention times within the milling chamber, flow rate variation, and equipment design limitation. Investigate variety development with bran layer component concentration considered. The goal being to increase within a given layer the concentration of valuable components such as vitamin E and oryzanol, or possibly other constituents of the rice bran layer such as rice bran saccharide or protein content.

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CHAPTER 4 ─ CONCLUSIONS AND RECOMMENDATIONS

Table 4.1 contains a summary for the parameters considered in this study by assigned category for both the Cheniere and Cypress varieties of rice. The parameter values included in Table 4.1 are for yield, degree of milling, transparency, whiteness, bran fraction, vitamin E, and oryzanol.

Conclusions

- 1. Laboratory values for yield showed little change across all time settings for both varieties of rice tested.
- 2. Pilot scale yield values decreased with increasing operational mill setting for both Cheniere and Cypress varieties.
- 3. Yield values for both rice varieties and both mill scales were highest at the low category of flow rate categorization.
- 4. Degree of milling measurements increased with increasing process time setting for the laboratory scale mill and with increasing operational mill setting for the pilot scale mill.
- 5. DOM data showed an increase for both varieties and both mill scales from the low to high categories.
- 6. Transparency and whiteness values followed similar patterns of increase from low to high flow rate category as DOM measurements displayed.
- 7. The amount of bran removed at the laboratory scale increased with process time setting.
- 8. Clear divisions of bran fraction by time setting were observed at the laboratory scale.
- 9. The amount of bran removed showed little variation between operational mill settings at the pilot scale.
- 10. Laboratory scale mill data presented by category showed distinct and increasing bran removal from the low to the high category.
- 11. Categorization of pilot scale data created separate divisions for each category for the Cypress variety, while the Cheniere variety data showed the same value for low and medium categories.
- 12. The highest levels of vitamin E measured occur at the 10 second setting for the laboratory mill and at the 3 setting for the pilot scale mill for both varieties of rice tested.
- 13. The low category exhibited the highest level of vitamin E for both varieties and mill scales, with the values trending down in value from the low to the high category.
- 14. Highest oryzanol values at the laboratory scale mill occurred at different time settings depending on variety.
- 15. The highest values of oryzanol at the pilot scale occurred at setting 3 for Cypress and Cheniere.
- 16. The low category for both varieties of rice and mill scales contained the highest level of oryzanol.
- 17. The amount of bran material removed increased with increasing process time setting at the laboratory scale.
- 18. At the pilot scale, bran removal occurred over a very narrow range of bran fraction, indicating little change between mill settings.
- 19. The lower settings for both scale mills have the higher or highest levels of antioxidant contained in a smaller amount of bran.
- 20. Operating at the low category would substantially reduce the quantity of bran requiring processing.

Recommendations

- 1. This work should be expended to the industrial scale.
- 2. Additional varieties should be tested for all scale mills.
- 3. Additional studies should be conducted at the low end of the pilot scale mill's operational mill setting scale to identify if higher antioxidant concentration results.
- 4. Examine the reason or reasons for the narrow range of bran fraction removed at the pilot scale, examining factors including: retention times within the milling chamber, flow rate variation, and equipment design limitation.
- 5. Investigate variety development to increase concentration of constituents within the bran layer.

Table 4.1, Values at Assigned Flow Rate Categories

Summary: Values at Flow Rate Categories

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APPENDIX A: PRE-EXPERIMENT PREPARATIONS

Sample Size

Prior to the experiment, several rough rice samples of approximately eleven kilograms (11.4 kg) were run through the pilot scale shelling unit. For each run, the amount (weight) of shelled rice produced from an initial half-sack sample was noted. As the weight of the shelled rice always exceeded nine kilograms, the shelled rice sample size was set at nine kilograms for each replicate to be milled.

Moisture Content

Rice bran is stabilized by heat treatment and removal of moisture to below three percent, followed by storage at low temperature (at least 0° C) ((Loeb et al, 1949, p. 739). A test was conducted to determine how long a period of drying was required for the moisture content of the bran removed to achieve a level below three percent moisture. The rice bran samples tested achieved equilibrium after only two hours of heating in a convection oven. Heat stabilization also halts the enzyme activity that deteriorates the quality of rice bran (Tao, 1989).

The procedural steps for the drying test conducted to determine the period of time required to achieve moisture removal:

- Three samples (5 gram) were selected from both varieties of rice.
- Samples were treated in a convection oven (PIC) at 95[°]C (approximately 200[°]F) for a period of 24 hours.
- Samples were weighed at five different times over the twenty-four hour test with values reported in Table 2.1.

After two hours, no change in the weight of any sample was observed. This observation justified a two hour drying time for sample stabilization as required for antioxidant content determination. Rice bran samples were heat stabilized and stored at (-18°C).

Moisture content affects the milling parameters for rice. Each variety can exhibited different properties at different moisture contents. Care was taken to maintain the same moisture content for the sacks of rice used for this experiment.

Measurements of moisture content of representative rough rice samples illustrated relative consistency of the initial moisture content of all the rough rice samples. The rough rice samples were stored at 0° C or below until about twelve hours before samples were processed. Initial moisture content, obtained with a grain analyzer (Dickey-John, Model GAC II), were recorded for both varieties in percentage values.

Table A.2, Percentage of mean moisture content of rough rice

Funnel Dimensions

A funnel was constructed from sheet metal to feed the shelled rice samples to the pilot scale mill's whitener or milling unit. The shape of the funnel was cut from sheet metal, bent into shape, and secured with pop-rivets.

Figure A.1, Custom Funnel Schematic

APPENDIX B: DATA TABLES

Table B.1, Summary Pilot Scale Summary Data for Cheniere

Table B.2, Pilot Scale Summary Data for Cypress

Table B.3, Laboratory Scale Summary Data for Cheniere

Table B.4, Laboratory Scale Summary Data for Cypress

Table B.5, Pilot Scale Yield for Cheniere

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Table B.6, Pilot Scale Yield for Cypress

Pilot Scale Mill Rice Variety: Cypress

Table B.7, Laboratory Scale Yield Date for Cheniere

Laboratory Scale Mill Rice Variety: Cheniere

Yield

Table B.8, Laboratory Scale Yield Data for Cypress

Laboratory Scale Mill Rice Variety: Cypress

Yield

Table B.9, Pilot Scale Degree of Milling Data for Cheniere

Table B.10, Pilot Scale Degree of Milling Data for Cypress

Table B.11, Pilot Scale Mill Transparency Data for Cheniere

Table B.12, Pilot Scale Transparency Data for Cypress

Table B.13, Pilot Scale Whiteness Data for Cheniere

Table B.14, Pilot Scale Whiteness Data for Cypress

Table B.15, Pilot Scale Bran Fraction Data for Cheniere

Table B.16, Pilot Scale Bran Fraction Data for Cypress

Table B.17, Pilot Scale Vitamin E Data for Cheniere

Table B.18, Pilot Scale Vitamin E for Cypress

Table B.19, Pilot Scale Oryzanol Data for Cheniere

Table B.20, Pilot Scale Oryzanol Data for Cypress

Laboratory Scale Mill Rice Variety: Cheniere										
Degree of Milling (Unpolished Milled Rice)										
Time (seconds)	Degree of Milling (DOM) Replicate			Mean Degree of Milling (DOM)	Standard Deviation of Mean					
		Ш	Ш							
5	45	49	45	46.33	2.31					
10	79	73	75	75.67	3.06					
15	97	91	95	94.33	3.06					
20	101	104	100	101.67	2.08					
25	107	110	110	109.00	1.73					
30	114	117	117	116.00	1.73					
35	122	118	114	118.00	4.00					
40	124	120	118	120.67	3.06					
45	128	128	127	127.67	0.58					

Table B.21, Laboratory Scale Degree of Milling Data for Cheniere

Table B.22, Laboratory Scale Degree of Milling Data for Cypress

Table B.23, Laboratory Scale Transparency Data for Cheniere

Table B.24, Laboratory Scale Transparency Data for Cypress

Laboratory Scale Mill Rice Variety: Cheniere									
Whiteness(Unpolished Milled Rice)									
Time (seconds)	Whiteness Replicate			Mean Whiteness	Standard Deviation of Mean				
		Ш	Ш						
5	29.3	30.0	29.7	29.30	0.53				
10	35.9	34.5	35.2	36.33	1.17				
15	39.3	37.9	38.0	39.13	0.61				
20	39.2	40.5	40.2	41.17	0.64				
25	40.7	41.7	42.5	41.57	0.93				
30	42.5	43.5	43.2	42.37	1.32				
35	44.1	43.6	42.5	44.13	0.40				
40	44.4	43.9	43.4	44.10	0.36				
45	45.8	45.3	46.0	45.33	0.49				

Table B.25, Laboratory Scale Whiteness Data for Cheniere

Table B.26, Laboratory Scale Whiteness Data for Cypress

Table B.27, Laboratory Scale Bran Fraction Data for Cheniere

Table B.28, Laboratory Scale Bran Fraction Data for Cypress

Table B.29, Laboratory Scale Vitamin E Data for Cheniere

Table B.30, Laboratory Scale Vitamin E Data for Cypress

Table B.31, Laboratory Scale Oryzanol Data for Cheniere

Table B.32, Laboratory Scale Oryzanol Data for Cypress

APPENDIX C: HPLC INFORMATION

HPLC was performed by Na Hua. a research assistant in the Biological and Agricultural Engineering Department, utilizing equipment in the Food Science Department at Louisiana State University. Hua is an experienced technician in HPLC techniques. She provided the below summary table for the tests conducted and a brief description of the process.

APPENDIX D: ORYZANOL INFORMATION

Table contains oryzanol data for Cheniere at the laboratory scale. The second replicate at the 30 second process time setting is an outlier and was excluded from the mean calculation of oryzanol for this time setting.

Laboratory Scale Mill Rice Variety: Cheniere Oryzanol									
Time (seconds)		Oryzanol (µg/g) Replicate		Mean of Oryzanol $(\mu g/g)$	Standard Deviation of Mean				
		Ш	Ш						
5	2546.20	2937.73	2529.65	2671.19	230.976				
10	2598.67	2724.38	2774.10	2699.05	90.416				
15	2511.67	2860.42	2320.26	2564.12	273.876				
20	2372.55	2418.15	2464.52	2418.41	45.982				
25	2135.70	2116.85	2157.62	2136.72	20.402				
30	2008.63	126.63*	2220.90	2114.77	150.098				
35	2014.66	1725.13	2291.29	2010.36	283.102				
40	1913.49	2346.09	2071.15	2110.25	218.932				
45	2003.99	1846.45	2217.22	2022.55	186.079				

Table 3.1, Laboratory Scale Mill Oryzanol Values for Cheniere

*Value excluded from mean calculations

VITA

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