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## Value-added processing of rice and rice by-products

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# VALUE-ADDED PROCESSING OF RICE AND RICE BY-PRODUCTS

A Dissertation

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  
Doctor of Philosophy

in

The Interdepartmental Program in Engineering Science

by  
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## **ABSTRACT**

World competition has encouraged United States rice farmers and rice mills to be efficient in farming and production practices. Efforts to augment economic competitiveness include development of new varieties, improvements in milling practices, and identification of uses for rice products and by-products. The research detailed in this dissertation adds to the body of knowledge in milling practices and identification of uses for rice bran. To improve the prediction of milled rice quality at industrial scale, correlations for milling quality among laboratory, pilot, and industrial scale mills were identified for Clearfield 161. Final industrial product whiteness was ten points higher than for polished rice at medium and high pilot scale settings. Jazzman, the first US-bred jasmine-type rice variety, was released by the LSU AgCenter Rice Research Station in 2009 to compete for a share of the aromatic rice market. Pilot scale evaluation of Jazzman's milling quality supported lab scale evaluation and provided additional data for milling optimization. With milling yields from 86 to 93%, Jazzman presented as a high-yield, good-milling aromatic long grain rice variety. A purple rice variety (line number MCR02-1576) was assessed for milling quality, and its bran for oil and anthocyanin concentration. Results showed a low milling recovery (<50%); low whiteness (<15%) values indicated pigment remained in the kernel. Anthocyanin concentration increased linearly across the entire bran layer. Oil concentration increased linearly across the inner bran layer with a mean of 22 percent. Processing the inner bran layer would maximize anthocyanin and oil recovery. As rice bran oil is a potential renewable energy source, the oil concentration across the bran layer of Jazzman, Clearfield 161, and Cocodrie were determined with hexane extraction and near infrared technology (NIT). Clearfield 161 had total oil concentration 1.83 times that of Jazzman and 2.11 times that of Cocodrie. Predictions of oil content across the bran layer were made from NIT

measurements and compared to hexane extraction results. Collectively this research indicates that value-added processing of rice and rice bran which optimizes milling yields and recovery of high-value components from the bran layer would favorably impact economic competitiveness.



# **CHAPTER 1. BACKGROUND AND JUSTIFICATION**

## **1.1 Introduction**

World competition has impacted the rice producing states in the United States; USA rice production accounts for approximately two percent of total global rice production and exceeded 640 million metric tons in 2007 (1, 2). Despite increased rice yield per acre of nearly 1134 kilograms (2500 pounds) over the last few decades (3), exports of rice from the United States have declined, showing a drop of over forty-three percent between 2004 and February 2009 (4). A thirteen percent decline is forecast for 2009 exports over 2008 exports, which will impact several regions of the country (5), as California, Missouri, Arkansas, Texas, Mississippi, and Louisiana are the six major rice producing states in the United States (3).

This competition forces United States rice farmers and rice mills to be efficient in farming and production practices. Efforts to augment competitiveness have included development of new varieties, implementation of new farming techniques, improvements in milling practices, and identification of uses for products and by-products. This research focused on enhancing rice quality by improving processing (milling), and on identifying alternative uses for rice bran, a by-product of the rice milling process.

Rice quality is characterized by standard parameters including milling recovery, head rice recovery, milling yield, degree of milling (DOM), transparency, and whiteness. Visual quality is quantified using industry measurements of whiteness, transparency, and degree of milling (6), which relate to the amount of bran removed and visual appearance of rice kernels. Milling yield refers to the weight of unbroken milled rice in ratio to the weight of milled rice. Head rice recovery measures whole kernel milled rice in ratio to the weight of rough rice processed (7). Millers use both milling yield and head rice recovery to evaluate overall rice kernel quality. The

United States Standards for Brown Rice for Processing (USDA) define a broken kernel as a kernel of rice that is less than three-fourths of a kernel for yield determination (8). DOM is a measure of the amount of the bran layer and germ removed by milling or processing (9). The amount of bran removed from shelled rice is a measure of the quality of the milled rice. To achieve a well-milled sample, the Standards Committee of the American Association of Cereal Chemists (2000) suggests a goal of a twelve percent weight reduction in the difference between shelled rice and milled rice weights during the milling process (10). Whiteness and transparency are measurements of physical rice kernel appearance (11).

Laboratory scale evaluation has traditionally provided milling quality information on new rice variety releases and in most cases has successfully predicted industrial scale performance (9). Different scale rice mills vary by capacity, processing format, and the number of milling chambers or breaks employed in the milling process. A McGill mill, a laboratory scale mill often used in testing, operates in batch format by removing bran from shelled rice in one chamber. LSU's pilot scale mill uses a stacked milling unit, with an abrasion chamber over a friction chamber, and continuous processing. Commercial mills operate in a continuous process using three or four milling units, referred to as milling breaks. Each break uses abrasion or friction to remove the bran layer (12). Milling studies at laboratory scale use samples of 125-150 grams to evaluate milling performance. The pilot scale mill uses a larger sample (11.5 kg) than the laboratory scale mill and a smaller sample than that required per test at industrial scale (900 kg) (9). Pilot scale milling provides a similar operating process to industrial scale, and when coupled with the smaller sample size required, makes testing at the pilot scale mill more logistically manageable and economically feasible for study (9).

Incidences of inaccuracy in predicting milling performance at industrial scale from laboratory scale testing have occurred (9,13). Pilot scale milling evaluation of Cocodrie addressed the problems encountered in scaling between laboratory and industrial scale for Cocodrie (9). The results of an optimization study conducted at the pilot scale provided useful information on the milling characteristics of Cocodrie which improved industrial scale milling of this variety (9). The success of this study to improve industrial scale milling performance led to this study's evaluation of Jazzman, a new rice variety released in 2009, and inspired questions about the predictive potential for milling characteristics of rice varieties among laboratory, pilot, and industrial scale mills.

The LSU AgCenter Crowley Rice Research Station has released 42 rice varieties which have enhanced rice crop value over its 100 year history (14). The development of a new rice variety progresses in a series of steps from small seed samples appropriate for laboratory scale testing to supplies large enough for release as seed stock (15). The US demand for jasmine-type rice has increased over the last decade (16); most of this demand is currently met with imports. Jasmine 85, an aromatic rice variety developed by the International Rice Research Institute, was released in Texas and has been grown on limited acreage in the southern United States. Jasmine 85 failed to meet consumer expectations due to gray coloring in the kernels and inferior aroma (16). In 1992, the LSU AgCenter Rice Research Station specialty rice breeding program was formed to develop aromatic rice varieties suitable for growing in Louisiana with the goal of competing for the aromatic rice niche market in the United States. Jazzman, the first US-bred jasmine-type rice was released by the LSU AgCenter Rice Research Station in 2009 (17), and is a long grain aromatic rice variety developed from a cross between an Arkansas variety, Ahrent, and an unreleased aromatic Chinese rice line 96a-8 (17). Jazzman exhibited good milling yield

and good milling quality in early laboratory scale screening tests (17). Jazzman has the potential to compete for a share of the aromatic rice market in the United States (17).

Line number MCR02-1576, a purple rice variety, was developed at the LSU AgCenter Rice Research Station in 1998 from a cross between Cypress and Hitan Kitan (C98-992) (2). Cypress, a long grain variety, was developed at the Rice Research Station in 1993; Hitan Kitan, a traditional purple variety, originated in Sri Lanka (2). The rice station provided a sample of line number MCR02-1576 for assessment of milling quality characteristics and evaluation of potential value-adding phytochemicals within the bran layer. Rice bran contains an array of health enhancing phytochemicals (7, 8).

Rice bran contains high quality oil and protein; cholesterol-lowering waxes; anti-tumor compounds like rice bran saccharide; antioxidants, including vitamin E and oryzanol; and in pigmented varieties, anthocyanins (9). Natural organic chromophores, such as anthocyanins, are used as pigments in electrical components, paints, optics, textiles (10), and foods (11). Anthocyanins exhibit antioxidant and anti-carcinogenic activities, hold promise as natural colorants, and show potential for photovoltaic applications. Anthocyanins have been used in cereal products as natural pigments and for their health benefits (10). Natural anthocyanins, extracted with acidified ethanol from several varieties of flowers, have found use as electron transfer compounds in dye-sensitive solar cells (9). Plant source anthocyanins are safer than rare metal oxides often used in solar cells, readily available from local sources, and easily extracted with inexpensive solvents (9).

Another component of interest in rice bran is oil. Rice bran contains oil in concentrations ranging from 10 to 23 percent by weight (12). Antioxidants, such as vitamin E and oryzanol, found in rice bran oil may help slow the onset of diabetes and Alzheimer's disease (13), and

appear to play a role in prevention of heart disease and multiple types of cancer (14). With increasing world demand for energy, plant sources of oil have been investigated as alternative energy sources. Rice bran oil, a renewable plant source of oil, has been investigated as stock material for biodiesel (18). Biodiesel has been made from rice bran oil by a variety of methods that have employed basic and acidic catalysts (15), metallic catalysts (16), and a biological catalyst, lipase (17). Kanitkar et al. (18) explored microwave-assisted extraction of rice bran oil and its effects on yield and oil properties. With methods of oil extraction and conversion of rice bran oil to biodiesel under study (15, 16, 17, 18), the identification of high oil varieties of rice and determination of the location of the highest concentration of oil in the bran layer would provide avenues to increase oil recovery.

Optimization of the feedstock for the conversion of rice bran oil to biodiesel provides a strategy for improving the economics of the conversion process. The identification of high oil concentration varieties of rice and determination of the location of the highest concentration of oil in the bran layer would provide avenues to increase oil recovery using a value-added processing approach. With oil concentration in the rice bran layer characteristically varying from 10 to 23 percent by weight (19), the identification of high oil content rice variety improves the potential for oil recovery by a ratio of 2.3 based on these literature values. Several methods for determination of oil concentration in grains or grain products or by-products have been reported in the literature. Traditionally, hexane has been used to extract oil from rice bran with the oil concentration reported as a weight percent of oil recovered to bran processed. Another approach to determining oil concentration in grains employs the use of near infrared technology (20). The identification of high oil concentration fractions of the bran layer could further increase the percentage of oil contained in a specific amount of bran material, and therefore the potential for

oil recovery. Rice varieties, with a high oil concentration in their bran layers, have potential as a renewable, plant source for oil, usable in nutritional, pharmaceutical, or a number of industrial applications.

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## **CHAPTER 2. MULTI-SCALE EVALUATION AND CORRELATION OF MILLED RICE QUALITY FOR CLEARFIELD 161**

### **2.1 Introduction**

Laboratory scale rice quality measurements have traditionally been utilized for predicting industrial scale mill performance. Research at industrial scale would require large amounts of rice, which is not available during variety development. Lab scale testing has provided a quick and easy method of predicting milled rice quality from a small sample of rice. Industrial scale mills have been slow to utilize the results from research conducted at laboratory scale as little evidence correlating different scale mills exists (1)

Laboratory scale mills operate as a batch process using friction milling; pilot and industrial scale mills operate as a continuous process using both abrasion and friction milling (1). Laboratory scale testing has usually successfully predicted performance of a rice variety at industrial scale; however, incidences of inaccuracy in predicting performance have occurred (2) (1). For example, we conducted an optimization study at pilot scale to address the problems encountered in scaling between laboratory and industrial scale for Cocodrie (2). Our results provided useful information on the milling characteristics of Cocodrie for industrial scale milling of this variety (2). This example of the pilot scale mill's potential for resolving issues inspired questions about the predictive potential for milling characteristics of rice varieties among laboratory, pilot, and industrial scale mills.

Rice quality is characterized by standard parameters including milling recovery, head rice recovery, milling yield, degree of milling (DOM), transparency, and whiteness. Milling recovery and head rice recovery are measurements based on the amount of rough rice processed, for total milled rice and unbroken rice kernels, respectively (3). Milling yield is determined by dividing



the weight of unbroken milled rice kernels by the total amount of rice milled (3). The United States Standards for Brown Rice for Processing (USDA) define a broken kernel as a kernel of rice that is less than three-fourths of a kernel for yield determination (4). With consumption of rice largely as milled rice, milling yield determines the value of the rice crop (5). The value of head rice is approximately twice that of broken kernels on an equal weight basis (5). DOM is a measure of the amount of the bran layer and germ removed by milling or processing (2). The amount of bran removed from shelled rice is a measure of the quality of the milled rice. To achieve a well-milled sample, the Standards Committee of the American Association of Cereal Chemists (2000) suggests a goal of a twelve percent weight reduction in the difference between shelled rice and milled rice weights during the milling process (6). Whiteness and transparency are measurements of physical rice kernel appearance (7).

Because of the economic impact of milled rice quality, this study sought to determine correlations for rice quality parameters among the different scales of rice mill. Milling one variety of rice at laboratory, pilot, and industrial scale would provide information for correlating quality measures among different rice mill scales. Specific study objectives were:

1. To measure rice quality parameters of milling yield, bran removal, whiteness, transparency, and DOM for a single rice variety, Clearfield 161, at laboratory, pilot, and industrial scale, and
2. To identify potential predictive correlations among laboratory, pilot, and industrial scale rice mills.

## **2.2 Materials and Methods**

Clearfield 161 rough rice for laboratory and pilot scale milling was provided by the LSU AgCenter's Rice Research Station. Industrial scale Clearfield 161 samples were obtained courtesy of a mill in southwest Louisiana. Samples were collected from all three mill scales at pre-selected times, settings, or locations.

Clearfield 161 was processed with nine treatments or sets of milling procedures. Treatments were defined by mill scale and mode of process control (or evaluation). Three treatments per mill scale were conducted in triplicate for settings corresponding to low, medium, and high levels of bran removed. Laboratory scale milling durations of L10, L25, and L40 (8), and pilot scale mill settings of 3, 5 and 9 (2) represent low, medium, and high levels of bran removed in respective order. At industrial scale, break 1, break 2, and break 3 represent the locations of low, medium, and high levels of bran removed (9). Table 2.1 presents treatment descriptions, numbered 1 to 9. Using Table 2.1, select a treatment number from 1 to 9, and move left to identify the milling scale, right to identify the time, setting, or location, and up to locate the category of bran removed as low, medium, or high. Treatment 4, 5, 6, and 9 results represent pilot and industrial scale milled rice products which were further processed by water polishing the rice kernels. Pilot scale treatments 4, 5, and 6 received the same amount of water polishing, and after polishing were labeled PP4, PP5, and PP6. The result of water polishing the milled rice product from treatment 9 was the industrial scale final polished rice product (IFP). Sample preparation, process steps, and process details by mill scale are detailed in the following paragraphs.

### **2.2.1 Sample Preparation**

The Clearfield 161 rough rice remained in cold storage (0°C) until required for experimentation, and was removed from the freezer twenty-four hours before shelling to permit the rice to reach ambient temperature (29°C) prior to milling. Industrial scale samples were also milled at ambient temperature (28-29°C). The rough rice supplied for laboratory and pilot scale milling had been cleared of foreign material prior to delivery to the researcher, and at industrial scale, dockage was conducted prior to milling.

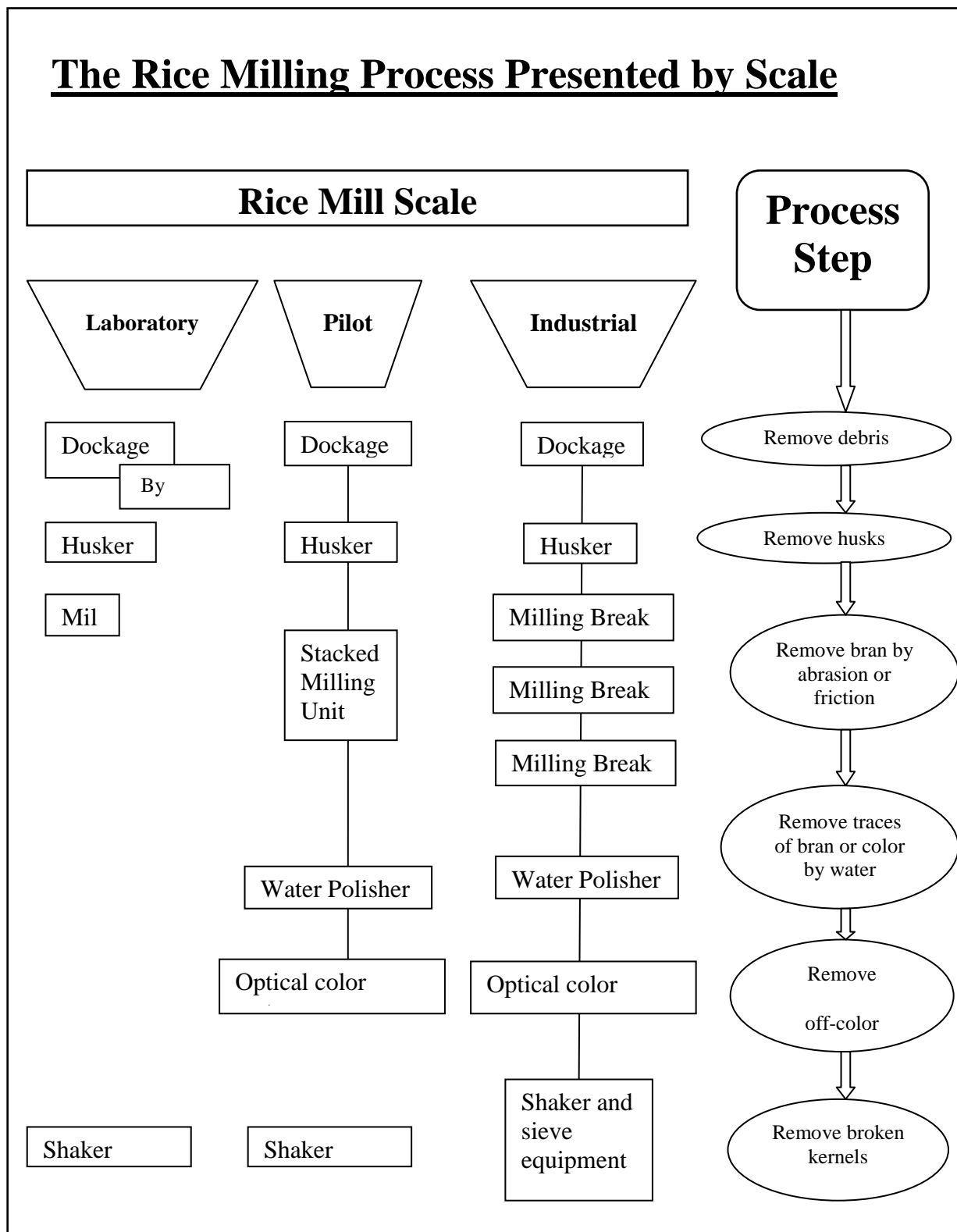
**Table 2.1. Treatment descriptions, treatments are numbered 1 to 9**

Mill Scale	Level (amount) of Bran Removed			Mode
	Low	Medium	High	
Laboratory	1			10
	2			25
	3			40
Pilot	4			3
	5			5
	6			9
Industrial	7			Break 1
	8			Break 2
	9			Break 3

### 2.2.2 Processing

Figure 2.1 presents the process steps for the three mill scales. Process steps for all three scales of rice mill are similar, but are performed by different methods or with different equipment. The first step, dockage, eliminates foreign material from the rough rice prior to shelling. Pieces of metal or stones are often found in the rough rice after harvesting and pose a risk of damage to shelling or husking equipment, which operates by passing the rough rice between a set of rollers. For all three mill scales, after dockage, rice is husked, and then milled. Following milling, rice is water polished and color-sorted at pilot and industrial scale. For laboratory and pilot scale mills broken kernels of rice are removed using a sorter table. At industrial scale, shaker tables and sieves are used to remove broken kernels of rice.

## The Rice Milling Process Presented by Scale



**Figure 2.1. Chart of three mill scales and process steps; lines connect equipment in continuous milling process**

#### **2.2.2.1 Lab Scale**

Rough rice was shelled (McGill, Model MS1, Grainman Machinery Co., Miami, Florida) and milled (McGill, Model 2, Brookshire, Texas) at time settings of 10, 25, and 40 seconds. One hundred and twenty five gram samples of shelled rice were milled; and, milled rice samples weighed and processed to remove broken kernels of rice. Broken kernels of rice were removed with a shaker table (Model 61-115-60, Grainman Machinery Co., Miami, Florida) using two sorter trays of sizes 10 (0.10 inch diameter indentations) and 12 (0.083 inch diameter indentations) at the top and bottom positions of the shaker table, respectively. Milled rice samples were weighed before and after processing with the shaker table to provide data to calculate milling yields.

#### **2.2.2.2 Pilot Scale**

Rough rice was processed with a pilot scale mill (Satake Engineering, Co., Tokyo, Japan) plant, which consisted of a shelling unit (Model GPS300A, Satake Engineering, Co., Tokyo, Japan), a milling unit (Model VAF10AM, Satake Engineering, Co., Tokyo, Japan), a water polisher (Model BA3AW, Satake Engineering, Co., Tokyo, Japan), and a color-sorter (Model GS3AA, Satake Engineering, Co., Tokyo, Japan). Rough rice samples of 11.5 kilograms were processed at pilot scale operational mill settings of 3, 5, and 9, which were selected to span the range of pilot scale settings of 0 to 10. Weights for shelled, milled, and polished rice were measured. Samples of milled and polished rice were processed with a shaker table (Model 61-115-60, Grainman Machinery Co., Miami, Florida) to remove broken kernels of rice.

#### **2.2.2.3 Industrial Scale**

Rough rice was processed at a commercial mill in southwest Louisiana. After dockage, the industrial scale mill consisted of a shelling unit (Model (HU10FTC2-1, Satake Engineering,

Co., Tokyo, Japan); a series of three or four milling units, known as milling breaks, (Buhler Vertical Millers, DSR-D62422, abrasive and friction units, Plymouth, MN); a unit to remove broken rice kernels (Model 3 UW 1FLDW, *Carter-Day* Company, Minneapolis, MN); a polishing unit (Buhler High Poly, no model number, Plymouth, MN); and a color sorting unit (Alpha Scan, Model SMII200, Satake Engineering, Co., Tokyo, Japan). Milled rice samples were collected from each milling break, and a polished rice sample was collected at process end. Samples of milled and polished rice were processed with a shaker table (Model 61-115-60, Grainman Machinery Co., Miami, Florida) to remove broken kernels of rice.

### **2.2.3 Measurements**

Milled rice samples were weighed before and after processing with the shaker table to provide data to calculate milling yields. Milling yield was calculated and reported as a percentage of the weight ratio of whole kernel milled rice to total milled rice (3). For each replicate at laboratory and pilot scale, the fraction of bran removed per mill setting was determined from weight ratio of the difference in shelled and milled rice to the weight of shelled rice. At industrial scale, percent bran removal had been set at each milling break by the mill operator (9). Degree of Milling (DOM), whiteness, and transparency readings were made using a Satake Milling Meter (Model MM1-D, Tokyo, Japan), an optical device using refraction or transmission of light for measurement of these parameters. DOM, whiteness, and transparency values were measured in triplicate for milled and polished rice samples; means were reported (2).

### **2.2.4 Statistical Analysis**

Table 2.2 presents the experimental design. The experiment was randomized and performed in triplicate for the nine selected treatments. Statistical analysis was performed with ANOVA, and Tukey mean comparisons. Predictive potential was assessed at a 5% significance

level (10). Table 2.3 presents treatments by treatment number and corresponding label. A capital letter indicated mill scale and a number indicated the control mode for each treatment.

**Table 2.2. Experimental Design**

Treatment Number	Milled Rice		Milled and Polished Rice*		
	Milling Yield	Percent Bran Removed	Whiteness	Transparency	Degree of Milling
1	----	----	----	----	----
2	----	----	----	----	----
3	----	----	----	----	----
4	----	----	----	----	----
5	----	----	----	----	----
6	----	----	----	----	----
7	----	----	----	----	----
8	----	----	----	----	----
9	----	----	----	----	----
Note: Dashes represent replicates					
* Three replicates for milled; three for polished					

**Table 2.3. Treatment number, treatment label**

Treatment Number	1	2	3	4	5	6	7	8	9
Treatment Label	L10	L25	L40	P3	P5	P9	B1	B2	B3
Letters represent mill scale: L for laboratory scale, P for pilot scale, B for industrial scale Numbers represent mode: seconds for laboratory scale, setting for pilot scale, milling break for industrial scale									

## 2.3 Results

For laboratory, pilot, and industrial scale, Table 2.4 presents results for milling yield and the percent of bran removed for milled rice, and displays whiteness, transparency, and DOM results for milled and polished rice. Blank spaces indicate when data was not collectable for the particular scale of rice mill. Values are indicated to plus or minus one standard deviation. A lowercase letter ‘a’ indicates information provided by rice miller at the request of the researcher.

**Table 2. 4. Mean values for milled rice milling yield, bran removed; milled and polished rice whiteness, transparency, and DOM**

Treatment Number	Milled Rice					Polished Rice		
	Milling Yield (%)	% Bran Removed	Whiteness	Transparency	DOM	Whiteness	Transparency	DOM
1	90.7 ± 0.2	7.8 ± 0.2	59.7 ± 0.3	2.7± 0.1	184.5 ± 0.7			
2	92.5 ± 1.9	12.6 ± 2.6	69.7 ± 0.8	3.3± 0.0	199.0 ± 0.0			
3	93.1 ± 0.7	14.9 ± 1.4	73.4 ± 1.5	3.7± 0.2	199.0 ± 0.0			
4	93.7 ± 1.7	13.8 ± 0.7	70.2 ± 1.3	3.4± 0.1	199.0 ± 0.0	65.6 ± 3.2	3.2 ± 0.2	199.0 ± 0.0
5	90.3 ± 1.5	15.5 ± 1.8	71.5 ± 1.0	3.5 ± 0.2	199.0 ± 0.0	70.8 ± 3.1	3.4 ± 0.1	199.0 ± 0.0
6	89.5 ± 0.6	15.6 ± 1.0	71.8 ± 1.0	3.4 ± 0.3	199.0 ± 0.0	71.1 ± 1.5	3.5 ± 0.1	198.0 ± 1.7
7	85.8 ± 1.4	5.6 <sup>a</sup>	57.2 ± 0.9	0.8 ± 0.1	164.5 ± 2.1			
8	87.6 ± 0.4	10.6 <sup>a</sup>	59.8 ± 0.6	1.1 ± 0.1	180.5 ± 0.7			
9	88.5 ± 1.2	12.5 <sup>a</sup>	65.7 ± 0.0	1.6 ± 0.0	199.0 ± 0.0	80.5 ± 1.7	2.7 ± 0.0	199.0 ± 0.0
Lower case superscript a indicates value provided by miller; blank spaces no data collectable at scale; treatment 4 two data points								



- Milled Rice

The Standards Committee of the American Association of Cereal Chemists (2000) suggests, for a well-milled sample, a weight reduction of twelve percent between shelled rice and milled rice sample weights (6). From the results of the three mill scales, a weight percent reduction of thirteen percent occurred with treatments 2, 4, and 9. Tukey mean comparisons indicated no statistical difference existed among these points which correspond to a laboratory scale milling duration of 25 seconds (L25), the pilot scale mill setting 3 (P3), and industrial scale milling break 3 (B3), respectively. Table 2.5 presents the weight percent bran removed results with plus or minus one standard deviation indicated for L25, P3, and B3.

**Table 2.5. Milled rice correlated numerical values with  $\pm$  one standard deviation indicated**

<b>Treatment</b>	<b>Number</b>	2	4	9
	<b>Label</b>	L25	P3	B3
<b>Mill</b>	<b>Scale</b>	Laboratory	Pilot	Industrial
	<b>Mode</b>	25	3	B3
<b>Quality Parameter</b>	<b>Bran Removed</b>	12.6 $\pm$ 2.6	13.8 $\pm$ 0.7	12.5a
	<b>Whiteness</b>	69.7 $\pm$ 0.8	70.2 $\pm$ 1.3	65.7 $\pm$ 0.1
	<b>Transparency</b>	3.3 $\pm$ 0.0	3.4 $\pm$ 0.0	1.6 $\pm$ 0.0
Lower case a indicates value determined by mill operator				

Siebenmorgen et al. (2006) milled different rice cultivars to the same DOM with a McGill No. 2 to compare additional milling characteristics (11) and found that different milling

durations were required to obtain the same DOM. Milling duration corresponds to the amount of bran removed from the rice kernel (3). This study used the weight percent of bran removed as the basis to compare additional milling characteristics (8). Employing percent bran removed as the basis resulted in the selection of treatments 2, 4, and 9 or L25, P3, and B3, respectively, as the points for comparison for whiteness and transparency of milled rice. No statistical difference was seen for whiteness or transparency values for L25, P3, and B3. Milled rice transparency values for treatments 2 and 4 were not significantly different, while treatment 9 was different than 2 and 4. Laboratory (L25) and pilot (P3) scale milled rice transparency values were not significantly different, but the industrial (B3) scale transparency value differed from laboratory and pilot scale measurements.

Milling yield determines the value of a rice crop, with the value of head rice approximately twice that of broken kernels on an equal weight basis (5). Mean milling yield (head milled over total milled) was 90 for all treatments, which compares well to Cypress, Cheniere, and Cocodrie values of 91, 87, and 88 percent, respectively (12). Statistical analysis of milling yield results for the nine selected treatments found no difference among treatments 1, 5, 6, 8, or 9. Table 2.6 presents milling yield results for these treatments, or by label, L10, P5, P9, B2 and B3, respectively. L10 represented the lowest level of bran removal at laboratory scale; P5 and B2 represented a medium level of bran removal; P9 represented the highest level of bran removal at pilot scale, and B3 was the last milling break before water polishing at industrial scale.

A study, comparing two commercial scale mills, one with a single break, and the second, a multi-break system, found lower milling yields with the single break system as bran removal (DOM) increased which probably resulted from differences in friction and abrasive milling (13).

**Table 2.6. Numerical values  $\pm$  one standard deviation for milling yield**

<b>Treatment</b>	<b>Number</b>	1	5	6	8	9
	<b>Label</b>	L10	P5	P9	B2	B3
<b>Mill</b>	<b>Scale</b>	Laboratory	Pilot	Pilot	Industrial	Industrial
	<b>Mode</b>	10	5	9	Break 2	Break 3
<b>Quality Parameter</b>	<b>Milling Yield</b>	$90.7 \pm 0.2$	$90.3 \pm 1.5$	$89.5 \pm 0.6$	$87.6 \pm 0.4$	$88.5 \pm 1.2$

We found a similar pattern of friction milling producing less kernel breakage than the combination of friction and abrasive milling. Our laboratory scale mill would correspond to the single break mill where friction milling dominates. Our pilot scale mill and the industrial scale mill would correspond to the multi-break mill with friction and abrasion milling occurring in a stacked milling unit or a series of milling breaks, respectively (13). Our results found higher milling yields at laboratory scale than at pilot or industrial scale with L10, exhibiting higher milling yields than P5 and P9, or B2 and B3.

- Polished Rice

Pilot scale and industrial scale mills employ a water polishing step to remove traces of bran or color from the rice kernels. Table 2.7 presents our results for polished rice at pilot and industrial scale. Polished rice results are limited to whiteness, transparency and degree of milling for pilot and industrial scale. Yield values could not be determined for industrial scale as the weight of rice before and after each process step was not obtainable. Pilot scale polished rice DOM measurements for the three tested settings showed no difference with polished industrial rice. Final product industrial scale transparency of 2.7 was lower than polished rice pilot scale

values. Industrial scale polished rice whiteness differed from pilot scale whiteness measurements; pilot scale values did not statistically differ among settings. A ten point difference existed between PP5 or PP9, and the final industrial scale product. The whiteness value for polished rice at both PP5 and PP9 was 71; and whiteness for polished rice at industrial scale was 81. The whiteness value at the highest pilot scale mill setting of 9, even after water polishing, was 10 points lower than for the polished product at industrial scale.

**Table 2.7. Polished Rice whiteness, transparency, DOM  $\pm$  one standard deviation**

<b>Treatment</b>	<b>Number</b>	4	5	6	9
	<b>Label (after polishing)</b>	PP3	PP5	PP9	IFP
<b>Mill</b>	<b>Scale</b>	Pilot	Pilot	Pilot	Industrial
	<b>Mode</b>	3	5	9	Final Product
<b>Quality Parameter</b>	<b>Whiteness</b>	65.6 $\pm$ 3.2	70.8 $\pm$ 3.1	71.1 $\pm$ 1.5	80.5 $\pm$ 1.7
	<b>Transparency</b>	3.2 $\pm$ 0.2	3.4 $\pm$ 0.1	3.5 $\pm$ 0.1	2.7 $\pm$ 0.0
	<b>DOM</b>	199.0 $\pm$ 0.0	199.0 $\pm$ 0.0	198.0 $\pm$ 1.7	199.0 $\pm$ 0.0

## 2.4 Conclusions

Predictive comparisons for milling quality among the three different mill scales were identified. Bran removal results were used to select correlation points for other tested parameters. A bran removal of 13 percent occurred at a 25 second laboratory milling duration (L25), a pilot scale setting of 3 (P3), and at industrial milling break 3 (B3). The laboratory milling duration of

25 seconds indicated a medium level of bran removal; the pilot scale setting of 3 represented a low level of bran removal; and industrial milling break 3 indicated a high level of bran removal. Milled rice whiteness values correlated well across scales at L25, P3, and B3. Milling yield correlated at a lab scale setting of 10, a pilot scale setting of 5 or 9, and at industrial scale milling breaks 2 or 3. The whiteness value for the final industrial scale product was ten points higher than the whiteness value at pilot scale settings of 5 or 9.

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## **CHAPTER 3. PILOT SCALE MILL CHARACTERIZATION OF JAZZMAN: FIRST US BRED AND RELEASED LONG GRAIN AROMATIC RICE VARIETY**

### **3.1 Introduction**

The LSU AgCenter Crowley Rice Research Station has released 42 rice varieties over its 100 year history (1). The development of a new rice variety progresses in a series of steps from small seed samples appropriate for laboratory scale testing to supplies large enough for release as seed stock (2). The US demand for jasmine-type rice has increased over the last decade (3). Most of this demand is being currently met by imports. Jasmine 85, an aromatic rice variety developed by the International Rice Research Institute, was released in Texas and has been grown on limited acreage in the southern United States. Jasmine 85 failed to meet consumer expectations due to gray coloring in the kernels and inferior aroma (3). In 1992, the LSU AgCenter Rice Research Station specialty rice breeding program was formed to develop aromatic rice varieties suitable for growing in Louisiana with the goal of competing for the aromatic rice niche market in the United States. Jazzman, the first US-bred jasmine-type rice was released by the LSU AgCenter Rice Research Station in 2009 (4).

Jazzman is a long grain aromatic rice variety that was developed from a cross between an Arkansas variety, Ahrent, and an unreleased aromatic Chinese rice line 96a-8 (4). It compares to Cypress in plant height and days to maturity with less susceptibility to blast and sheath blight than Cheniere (4). Jazzman averaged 7803 kilograms per hectare in yield trials over five years in five states, which compares favorably with two non-aromatic long grain varieties, Cypress and Cheniere (4). Cypress and Cheniere had mean yield values of 7841 kilograms per hectare and 8903 kilograms per hectare, respectively (5). Jazzman exhibited good milling yield and good milling quality in early laboratory scale screening tests (4). Head rice recovery (weight ratio of

head milled to rough rice processed) for Jazzman was 63.6 percent, which falls in the range of the values for Cheniere (63.1) and Cypress (64.3) (5). Initial release documentation, the source for the information on Cypress, Cheniere, and Jazzman, did not include values for DOM, whiteness, and transparency (4; 5). Because these values are important quality indicators, we measured them at the pilot scale.

Laboratory scale evaluation has traditionally provided milling quality information on new rice variety releases, and in most cases, successfully predicted industrial scale performance (6). Different scale rice mills vary by capacity, processing format, and the number of milling chambers or breaks employed in the milling process. The standard laboratory mill used in testing, a McGill mill, operates in batch format by removing bran from shelled rice in one chamber. Our pilot scale mill uses a stacked milling unit, with an abrasion chamber over a friction chamber, and continuous processing. Commercial mills operate in a continuous process using three or four milling units, referred to as milling breaks. Each break uses abrasion or friction to remove the bran layer (7). Milling studies at laboratory scale use samples of 125-150 grams to evaluate milling performance. The pilot scale mill uses a larger sample (11.5 kg) than the laboratory scale mill and a smaller sample than that required per test at industrial scale (900 kg) (6). Pilot scale milling provides a similar operating process to industrial scale, and when coupled with the smaller sample size required, makes testing at the pilot scale mill more logistically manageable and economically feasible for study (6). Milling quality assessment for laboratory, pilot, and industrial scale includes evaluation of grain appearance, and measurement of milling yield and head rice recovery. Visual quality is quantified using industry measurements of whiteness, transparency, and degree of milling (8), which relate to the amount of bran removed and visual appearance of rice kernels. Milling yield refers to the weight of unbroken



milled rice in ratio to the weight of milled rice; head rice recovery measures whole kernel milled rice in ratio to the weight of rough rice processed (9). Millers use both milling yield and head rice recovery to evaluate overall rice kernel quality.

In a pilot scale study milling Cocodrie, Hua et al. (2006) examined processing parameters of milling recovery, wholeness, transparency, whiteness, and degree of milling (DOM) as a function of roll gap size in husking, feed volume or flow rate during milling, and water velocity during polishing. Hua et al. (2006) determined that flow rate was the only significant (controlling) parameter (6). Optimal mill settings for milling of Cocodrie at pilot scale were determined, and utilized to improve milling quality at the industrial scale (6).

Processing Jazzman at different flow rates provided additional information on the variety's milling characteristics. This characterization of Jazzman could help ensure success at industrial scale. Specific study objectives were:

1. To measure the percent of bran removed at selected pilot scale mill operational settings,
2. To determine values for quality assessments (head rice recovery, whiteness, transparency, and degree of milling) at selected pilot scale mill operational settings, and
3. To identify the optimal range of mill settings for Jazzman.

## **3.2 Materials and Methods**

### **3.2.1 Sample Preparation**

Jazzman provided by the LSU AgCenter Rice Research Station was stored in a freezer (0°C) until the day before processing. A period of 24 hours allowed the rice to achieve ambient temperature (29°C) before milling. Enough rice was removed from the freezer for testing to be conducted in triplicate.

### **3.2.2 Processing**

Rice was processed using a pilot scale milling plant (Satake Engineering Co. Tokyo, Japan) which operates as a continuous process and consists of a husker (Model GPS300A), a mill (Model VAF10AM), a wet polisher (Model BA3AW), and a color sorter (Model GS3AA). Rough rice samples of 11.5 kilograms were processed at three selected pilot scale operational settings. Milled and polished rice samples were collected. Milled rice refers to rice processed with the milling or whitening unit (Model VAF10AM). Polished rice has been further processed with the water polisher (Model BA3AW).

### **3.2.3 Measurements**

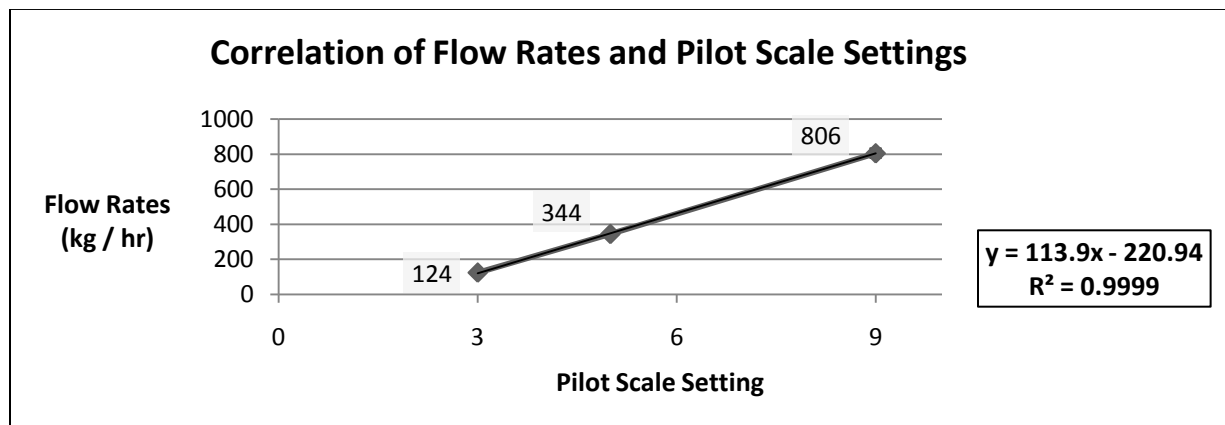
#### **3.2.3.1 Flow Rate**

Flow rates were determined at the milling unit by weighing a volume of milled rice that was collected for a recorded time. From triplicate measurements, mean flow rates were reported in kilograms per hour for pilot scale mill settings of 3, 5, and 9. The settings selected (3, 5, and 9) correspond to low, medium, and high flow rates used by Hua et al. (2006) in an optimization study of Cocodrie. Figure 3.1 presents the relationship for milled rice between pilot scale mill setting and flow rate in kilograms per hour. The flow rate increased with increasing pilot scale mill setting from 124 to 806 kilograms per hour and exhibited a linear relationship with mill setting. The flow rate of milled rice approximately doubled in value between successive pilot scale mill settings.

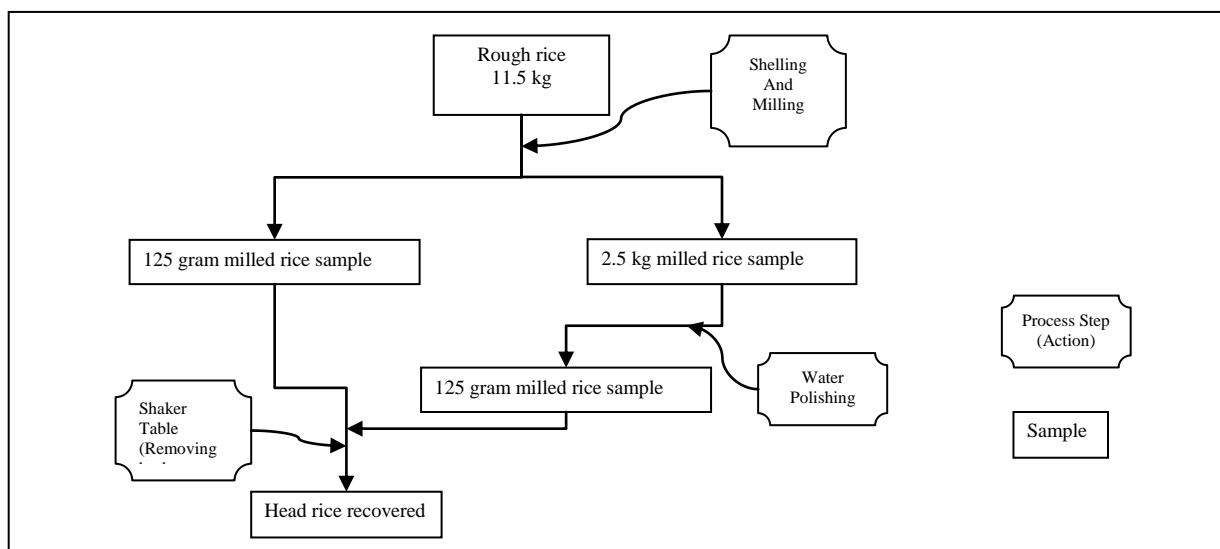
#### **3.2.3.2 Bran Removed and Milling Quality**

Nine rough rice samples of 11.5 kilograms were shelled and milled with weight measured after each process step. Figure 3.2 shows the sample collection process. Shelled rice samples of 125 grams and 2.5 kilograms were collected from each replicate processed. The 2.5 kilogram

sample was further processed through the water polisher and reweighed. From the polished rice, samples of 125 grams were collected. A shaker table (Model 61-115-60, Grainman Machinery Co., Miami, Florida) was used to remove broken kernels of rice from the 125 gram milled and polished rice samples using trays of sizes 10 (0.10 inch diameter indentations) and 12 (0.083 inch diameter indentations) at the top and bottom positions of the shaker table, respectively.



**Figure 3.1. Regression flow rate and pilot scale setting; Setting 9 two data points**



**Figure 3.2. Chart of sample collection**

Milling yield was reported as a weight percentage of milled (or polished) rice to the shelled rice processed (9). Head rice recovery was determined as a weight percent of unbroken kernels to the weight of the rough rice sample processed for milled and polished rice (9). The

amount of bran removed for each mill setting was determined from the difference between the weights of shelled and milled rice. Bran removal was reported as a percentage based on the weight ratio of bran removed to the weight of shelled rice processed. During milling, whiteness, transparency, and degree of milling were measured for milled and polished rice samples in triplicate with a Satake milling meter (model MM-1D, Tokyo, Japan) (6); the mean of the three readings was reported. Based on milling quality results, the best operational conditions were determined.

### **3.2.4 Statistical Analysis**

Experiments were completely randomized. Three replicates were performed for tested pilot scale settings. Statistical analysis at a 5 percent significance level was performed with ANOVA and Tukey mean comparisons using XLSTAT-Pro (2009) (10).

## **3.3 Results**

### **3.3.1 Bran Removed and Milling Quality**

Figure 3.3 displays bran removal and milling quality results for milled and polished rice by operational setting. The amount of bran removed by milling was reported for milled rice; the total amount of bran removed by milling and polishing was reported under the polished rice category. Table 3.1 presents numerical values with one standard deviation indicated for bran removed, head rice recovery, DOM, whiteness and transparency.

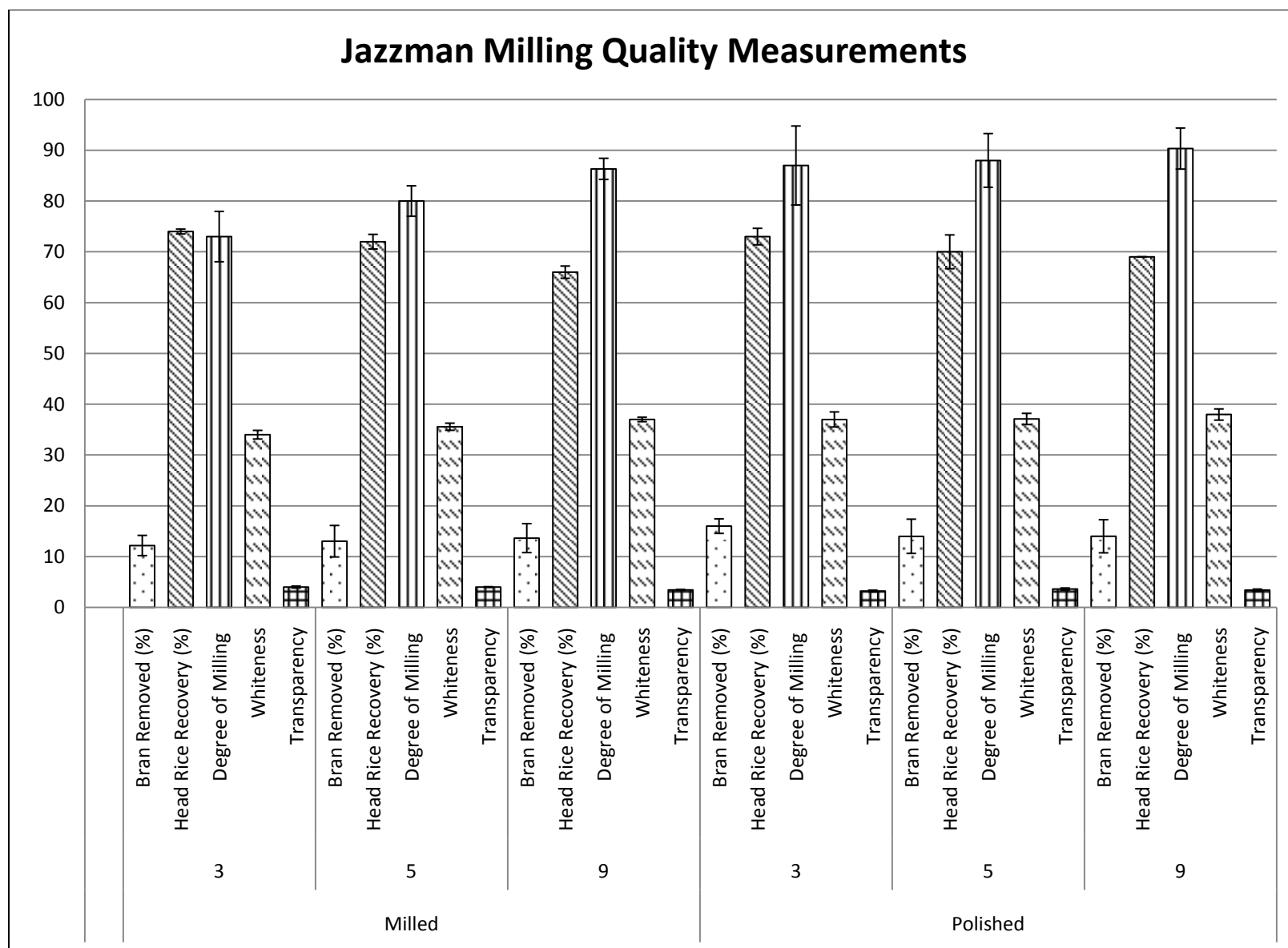
### **3.3.2 Bran Results**

The percent of bran removed at a selected mill setting, the degree of milling (DOM), and whiteness values increase with increasing flow rates for milled rice. No difference in the mean amount of bran removed was observed between successive mill settings for milled rice, indicating that bran is easily removed from the kernel for this variety. The percent of bran

removed ranged from 12 to 14 for milled rice, and from 14 to 16 for polished rice. Water polishing removed 3.4, 1.0, and 0.6 percent bran at mill settings of 3, 5, and 9, respectively. The Standards Committee of the American Association of Cereal Chemists in 2000 recommended a twelve percent reduction in weight between shelled rice and milled rice samples for a well milled sample (11). With all samples meeting well milled standards, the goal becomes to maximize whole kernels while increasing whiteness.

### **3.3.3 Head Rice Recovery and Milling Yield**

Jazzman milling yield and head rice recovery compare well to non-aromatic and aromatic long grain rice varieties. Head rice recovery decreases with increasing flow rate for milled and polished Jazzman due to the increase in friction milling at higher flow rates. The mean head rice recovery for milled rice on a rough rice basis was 70.9; the mean head rice recovery for polished rice on a rough rice basis was 70.8. No significant difference existed between mean values for milled and polished head rice recovery. Laboratory scale evaluation found a head rice recovery of 63.6 percent for Jazzman (4). Jazzman's head rice recovery (4) compares well with head rice recovery values of Cypress and Cheniere, which are considered good milling varieties of rice with head rice recovery values of 64.3 and 63.4, respectively (5). Jazzman's mean pilot scale head rice recovery value of 71 was higher than the laboratory measurement of 63.6 due to process differences. Laboratory scale mills operate in a batch format with all milling accomplished in one step; pilot scale mills operate by removing bran in a series of steps (7). Chen et al. (1998) found more rice was broken in a single break system than a multiple break system for any DOM level (7). They observed progressively decreasing head rice recovery with increasing DOM for the single break system, but for the multiple-break milling mill, most of the broken kernels were seen early in milling (7).



**Figure 3.3. Jazzman milled and polished rice bran and milling quality measurements; means  $\pm$  one standard deviation**

**Table 3.1. Summary of rice quality measurements with one standard deviation indicated**

Rice Quality Measurements		Pilot Scale Setting		
		3	5	9
Bran Removed (%)	Milled Rice	12.2 ± 2.0a	13.0 ± 3.1a	13.6 ± 2.9a
	Polished Rice	3.4 ± 1.0a	1.0 ± 0.3b	0.6 ± 0.5b
Head Rice Recovery (%)	Milled Rice	74 ± 0.5a	72 ± 1.4a	66 ± 1.2b
	Polished Rice	73 ± 1.6a	70 ± 3.3a	69 ± 0.0b
Degree of Milling (DOM)	Milled Rice	73 ± 4.9a*	80 ± 3.0a	86 ± 2.1b
	Polished Rice	87 ± 7.8j*	88 ± 5.3j	90 ± 4.0j
Whiteness	Milled Rice	33.5 ± 0.8a*	35.6 ± 0.7a	37.0 ± 0.4b
	Polished Rice	37.4 ± 1.5a*	37.1 ± 1.1a	38.0 ± 1.1a
Transparency	Milled Rice	3.7 ± 0.2a*	3.5 ± 0.1a	3.4 ± 0.1b
	Polished Rice	3.2 ± 0.2a	3.6 ± 0.2a	3.4 ± 0.2a
The same lower case letter across a row indicates no statistical difference; (*) indicates one less data point				

Mean milling yield (head milled over total milled) was 90 percent for Jazzman. Jazzman values compared well to the non-aromatic, long-grain varieties Cypress, Cheniere, and Cocodrie with milling yields of 91, 87, and 88 percent respectively (5). Islam et al. (2003) studied milling quality of two Bangladesh long grain aromatic varieties using thirty kilogram rough rice samples (pilot scale) (12). Milling yields for the two tested aromatic varieties were 90.4 and 84.1 percent with head rice recovery measured as 60.2 and 60.1, respectively (12). Jazzman's milling yield of 90 was in the range for the two Bangladesh aromatic varieties, and Jazzman's head rice recovery of 71 exceeded the Bangladesh varieties head rice recoveries.

### **3.3.4 Quantification of Visual Appearance**

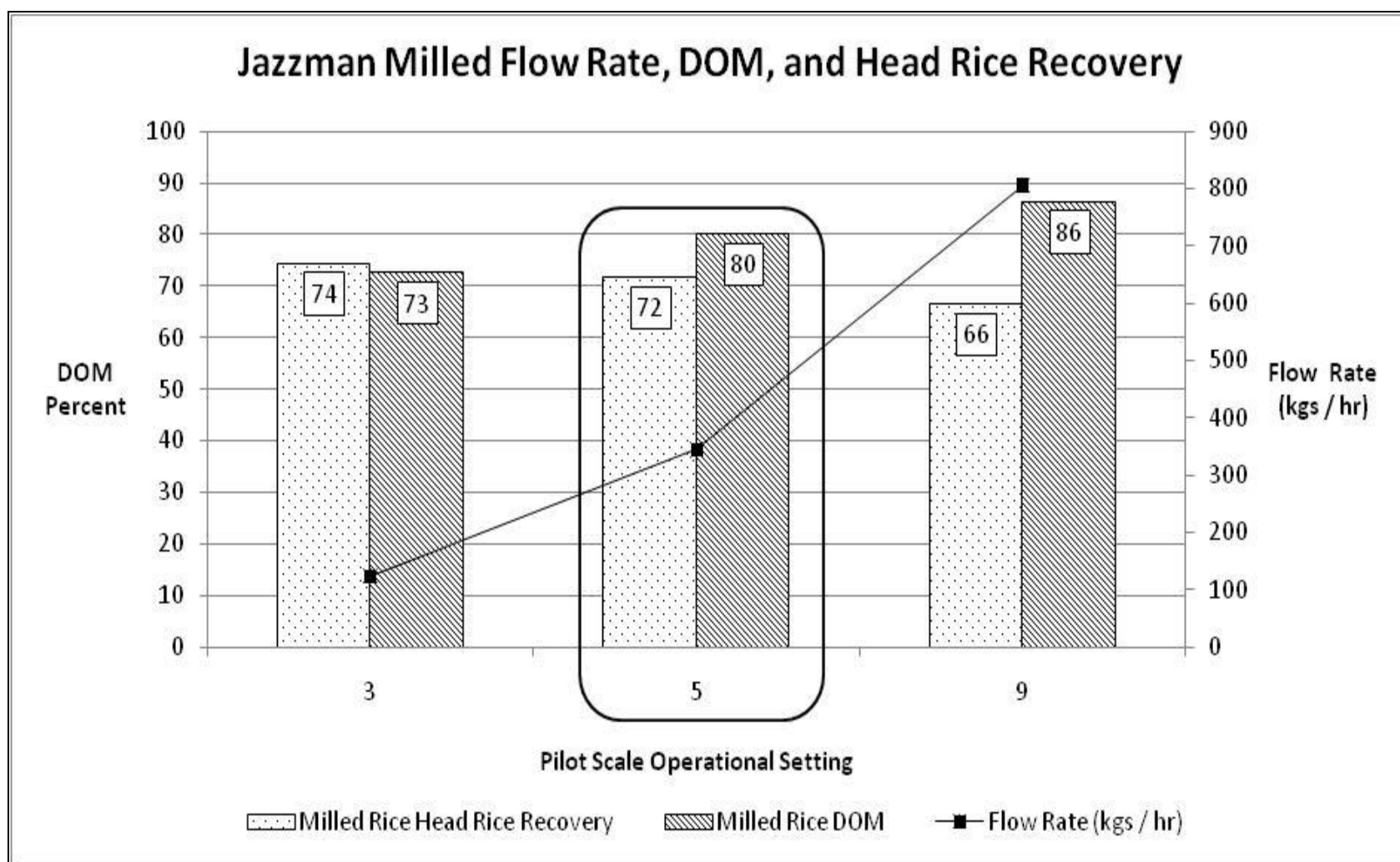
Measurements for DOM, whiteness, and transparency indicate the appearance quality of rice kernels. Literature review found few references including DOM, whiteness, or transparency values. A laboratory scale study of medium rice determined mean whiteness values of 41.5 to 45.7, dependent on rice variety and environmental conditions (13). Our study found whiteness values of 35 for milled and 37 for polished rice. The few points of difference in whiteness values observed between studies were accounted for by the level of processing, by measurement of different rice varieties, by differences in rough rice quality, or by a difference in milling scale. Milled and polished rice DOM values for our study were 80 and 88, respectively. DOM and whiteness values increased with flow rate; transparency values exhibited little change with flow rate. Tukey comparisons of mean values for DOM and whiteness found differences between milled and polished rice values. The polished rice DOM of 88 falls within the range of DOM values expected at commercial scale. Commercial mills expect a DOM in the range of 85 to 95 (14). Transparency values ranged less than 0.4 percent and exhibited no significant differences.



### 3.3.5 Optimal Milling Performance

Figure 3.4 presents milled rice DOM and head rice recovery as a function of mill setting. The final decision of operational flow rate affects the head rice recovery and kernel appearance. At laboratory scale, head rice recovery and DOM exist in an inverse relationship (14). Our findings at pilot scale display the same general pattern of higher DOM with lower head rice recovery. Increasing flow rate increases the amount of bran removed and the number of broken kernels, establishing an inverse relationship which must be considered when selecting an operational range for flow rate.

Industrial scale rice millers apply information from multiple steps, or breaks, along the milling process to optimize the milling process by balancing increasing broken rice kernels with increasing DOM and whiteness (6). For milled rice, Jazzman exhibited minimal differences in head rice recovery and DOM between flow rates. Head rice recovery for pilot mill setting 9 was lower than for settings 3 or 5. Head rice recovery values at 3 and 5 exhibited no statistical difference. Milled rice DOM values exhibited no difference between 5 and 9. Combining statistical analysis, setting 5 optimizes head rice recovery and DOM. Milled rice at setting 5 has head rice recovery of 72 and a DOM of 80. The head rice recovery exceeded values for Cypress and Cheniere with head rice recoveries of 64 and 63, respectively. Although the DOM value for milled rice at setting 5 fails to meet industry expectations for DOM of 85 to 95 (14), after water polishing, the DOM at setting 5 was 88, meeting commercial expectations. Head rice recovery was 69, also acceptable at commercial level. In summary, milling at pilot scale setting 5, which correlated to a medium flow rate for the pilot scale mill, optimized head rice recovery and DOM for the long grain aromatic rice variety, Jazzman.



**Figure 3.4. Milled rice DOM, head rice recovery; means  $\pm$  one standard deviation; rounded-rectangle indicates optimal**

### 3.4 Conclusions

Jazzman yields and milling quality were comparable to two commercially successful non-aromatic varieties grown in the same geographic area and to two long grain aromatic varieties grown in Bangladesh (12). Flow rates and pilot scale settings selected followed a linear relationship with the rate of flow approximately doubling between successive mill settings. DOM and whiteness increased with flow rate increase; head rice recovery decreased with flow rate increase. The small amount of additional bran removed by water polishing resulted in whiteness values exhibiting no differences between mill settings for polished rice. Water polishing had minimal effect on head rice recovery, but improved the degree of milling of the final product from 80 for milled rice to 88 for polished. After water polishing, the DOM at setting 5 was 88, meeting commercial expectations; head rice recovery was 69, acceptable at commercial level. Jazzman presented as a high yield, good milling aromatic rice variety. Pilot scale rice milling indicated that Jazzman is a commercially viable rice variety.

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# **CHAPTER 4. LAB SCALE CHARACTERIZATION OF A PURPLE RICE VARIETY: EXAMINATION OF MILLING QUALITY AND DETERMINATION OF ANTHOCYANIN AND OIL CONCENTRATIONS ACROSS THE BRAN LAYER**

## **4.1 Introduction**

The LSU AgCenter Crowley Rice Research Station has developed 42 rice varieties which have enhanced rice crop value over its 100 year history (1). Varieties exhibiting desired traits are selected and bred to enhance these traits; the quality of milled rice samples from test varieties impacts this selection process (1). The development of a new rice variety progresses in a series of steps from small seed samples appropriate for laboratory scale testing to supplies large enough for release as seed stock (1). From the Rice Research Station, we were provided with 20 kilograms of this variety for assessment of milling quality characteristics and evaluation of potential value-adding phytochemicals within the bran layer. Line number MCR02-1576 was obtained from a 1998 cross between Cypress and Hitan Kitan (C98-992) (2). Cypress, a long grain variety, was developed at the Rice Research Station in 1993; Hitan Kitan, a traditional purple variety, originated in Sri Lanka (2).

Milling quality refers to physical characteristics of the rice kernel and is characterized by determining degree of milling (DOM), whiteness, transparency, and milling recovery at specific mill settings. DOM, whiteness, and transparency indicate the quality of the milled rice kernels in terms of visual appearance. Milling recovery indicates quality in terms of whole rice kernels (head rice) produced from milling; head rice milling recovery was measured as the weight percent of the head rice over the rough rice processed. DOM, whiteness, and milling recovery measurements for non-pigmented rice varieties have been reported in the literature.

Commercially for non-pigmented varieties, DOM values are expected to range between 85 and

95 (3). Pan et al. (4) measured mean whiteness values ranging from 42.3 to 43.8, dependent on rough rice quality. The head milling recovery rate for two non-pigmented, good-milling rice varieties, measured near 64 percent. Cypress, the non-pigmented parent variety of the purple rice under study, measured 63.8 (5), and Cheniere, another LSU AgCenter variety release, had a head rice recovery of 63.6 percent (5). One article addressing pigmented rice milling quality was located in the literature. Patindol et al. (2005) measured head rice recovery values for 16 red rice samples; results varied between states and within states for red rice samples tested; head milling recovery values ranged from 51.3 to 64.3 weight percent based on rough rice (6). Five of the samples tested had purple-black hulls with all but one variety displaying head milling recovery less than 60 percent (6). The purple-black hulled varieties had whiter milled rice than other tested varieties, except the two cultivated varieties (6). DOM, whiteness, and transparency values were not reported.

Rice bran contains an array of health enhancing phytochemicals (7, 8). Rice bran contains high quality oil and protein; cholesterol-lowering waxes; anti-tumor compounds like rice bran saccharide; and antioxidants, including vitamin E and oryzanol; and in pigmented varieties, anthocyanins (9). Natural organic chromophores, such as anthocyanins, are used as pigments in electrical components, paints, optics, textiles (10), and foods (11). Anthocyanins exhibit antioxidant and anti-carcinogenic activities, hold promise as natural colorants, and show potential for photovoltaic applications. Anthocyanins have been used in cereal products as natural pigments and for their health benefits (10). Natural anthocyanins, extracted with ethanol from several varieties of flowers, have found use as electron transfer compounds in dye-sensitive solar cells (9). Plant source anthocyanins are safer than rare metal oxides often used in solar cells, readily available from local sources, and easily extracted with inexpensive solvents (9).

Rice bran contains oil in concentrations ranging from 10 to 23 percent by weight (12). Antioxidants, such as vitamin E and oryzanol, found in rice bran oil may help slow the onset of diabetes and Alzheimer's disease (13), and appear to play a role in prevention of heart disease and multiple types of cancer (14). With increasing world demand for energy, plant sources of oil have been investigated as alternative energy sources. Biodiesel has been made from rice bran oil by a variety of methods that have employed basic and acidic catalysts (15), metallic catalysts (16), and a biological catalyst, lipase (17). Kanitkar et al. (18) explored microwave-assisted extraction of rice bran oil and its effects on yield and oil properties. With methods of oil extraction and conversion of rice bran oil to biodiesel known (15, 16, 17, 18), the identification of high oil varieties of rice and determination of the location of the highest concentration of oil in the bran layer would provide avenues to increase oil recovery.

Value-added processing of rice bran to increase the recovery of anthocyanins or oil potentially impacts human health, energy concerns, and environmental issues. Processing the bran layer into divisions permits the use of the separate fractions for recovery of the high-value component of interest. This selective approach optimizes the process of extraction by using the fraction of bran containing a higher concentration of the high-value component while reducing the total amount of bran requiring processing (19).

The objectives of our study were:

1. To determine milling quality parameters of line number MCR02-1576 at lab scale,
2. To measure degree of milling, whiteness, and transparency measurements with a milling meter,
3. To determine the anthocyanin concentration gradient across the bran layer of line number MCR02-1576, and
4. To determine the oil concentration across the bran layer of line number MCR02-1576.

## **4.2 Materials and Methods**

### **4.2.1 Sample Preparation**

Rough rice was supplied by the LSU Agricultural Center (Louisiana Rice Research Station, Crowley, LA) and stored at 0°C until testing. Before processing, rice was removed from cold storage and allowed 24 hours to equilibrate to the ambient temperature of the milling laboratory (29°C). Mean moisture by weight measured 14.2 percent for rough rice samples tested.

### **4.2.2 Processing**

Rough rice samples required several processing steps to obtain a bran sample at laboratory scale. The first step involved removing the outer husk layer with a McGill Sheller (Model MS1, Grainman, Florida). After the shelling step, shelled rice samples of 125 grams were milled with a McGill mill (Model No. 2, Brookshire, Texas) at time settings of 10, 25, 40, 45, 55, and 65 seconds. Broken kernels were removed from the milled rice sample with a shaker table (Model 61-115-60, Grainman Machinery Co., Miami, Florida) using two sorter trays of sizes 10 and 12 at the top and bottom positions of the shaker table, respectively. Materials produced from replicate runs at each processing step were collected and weighed. Bran from each replicate was collected and stored (-18°C) until extraction and determination of anthocyanin and oil concentrations. Samples of milled rice materials were assessed for quality.

### **4.2.3 Measurements**

#### **4.2.3.1 Milling Quality Assessment**

Milling quality was assessed at operational time settings of 10, 25, and 40 seconds by determining values for DOM, whiteness, transparency, milling recovery, and the amount of bran removed at each setting. These settings were selected because they correspond to low, medium,



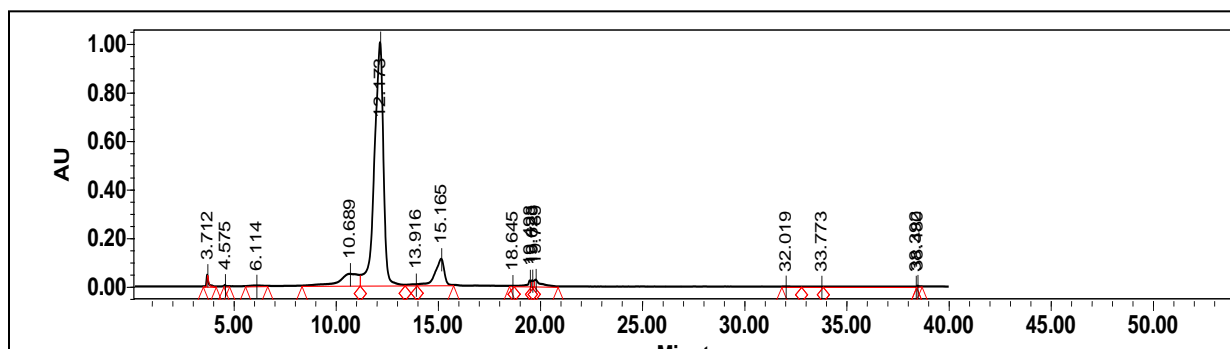
and high bran removal rates from previous research (20). DOM, whiteness, and transparency were measured with a milling meter (Model MM-1D, Satake Engineering Co., Tokyo, Japan). Milling recovery, a milling quality measure reported in percent, was defined as the weight of whole unbroken milled kernels divided by the weight of the rough rice processed (21). The amount of bran removed for each milling length was reported as a weight fraction determined from the bran removed divided by the shelled rice processed.

#### **4.2.3.2 Anthocyanin and Oil Extractions and Concentration Evaluation**

Oil and anthocyanin concentrations across the bran layer were determined for bran samples collected at 10, 25, 40, 45, 55, and 65 seconds. Before anthocyanin extraction, lipids were removed from the bran samples using a two-solvent extraction process (22). Our method was adapted from Jang and Xu (23), who used hexane to extract lipids and methanol to extract anthocyanins. We used ethanol instead of methanol as the anthocyanin extraction solvent based on its polarity index (24), GRAS status (25), and industry guidelines (26). Half-gram samples of rice bran were placed in test tubes with 3 milliliters of hexane and incubated at 60°C for 30 minutes in a water bath. After incubation, the test tubes were centrifuged (1500 rpm, 5 minutes), the solvent decanted, and saved for oil concentration determination. The remaining hexane was evaporated from the bran residue prior to the addition of 3 milliliters of ethanol (22). After incubation for 30 minutes in a water bath at 60°C, the solvent portion was used to determine anthocyanin concentration with high pressure liquid chromatography. The hexane was evaporated from the decanted solution and the remaining oil weighed. Oil concentration was reported as a weight fraction of oil extracted to bran processed.

HPLC analysis was conducted with a system comprised of a Supelco (Bellefonte, PA) Discovery C18 column (inside diameter 3mm, length 25 cm), a Waters 2690 separation module,

a 996 photodiode array detector, and a Millennium 32 chromatography manager. The mobile phase used a constant flow rate of 0.8 milliliters per minute with a composition of A: 0.4 % trifluoroacetic acid (TFA) in water and B: acetonitrile, with a percentage of A: 0.4% TFA in water ramped from 100% to 55% in 45 minutes. Figure 4.1 presents an example of the graphs obtained at 520 nm with high pressure liquid chromatography with solution obtained from ethanol extraction. From a standard curve, the concentration of selected peaks was determined, and the total anthocyanin content calculated by summing the concentration of anthocyanins from peaks located at 10, 15, and 22 second elution times. Anthocyanin values were reported in micrograms per gram of rice bran extracted with solvent.



**Figure 4.1. HPLC graph of anthocyanin concentration in bran removed with a 40 second milling duration**

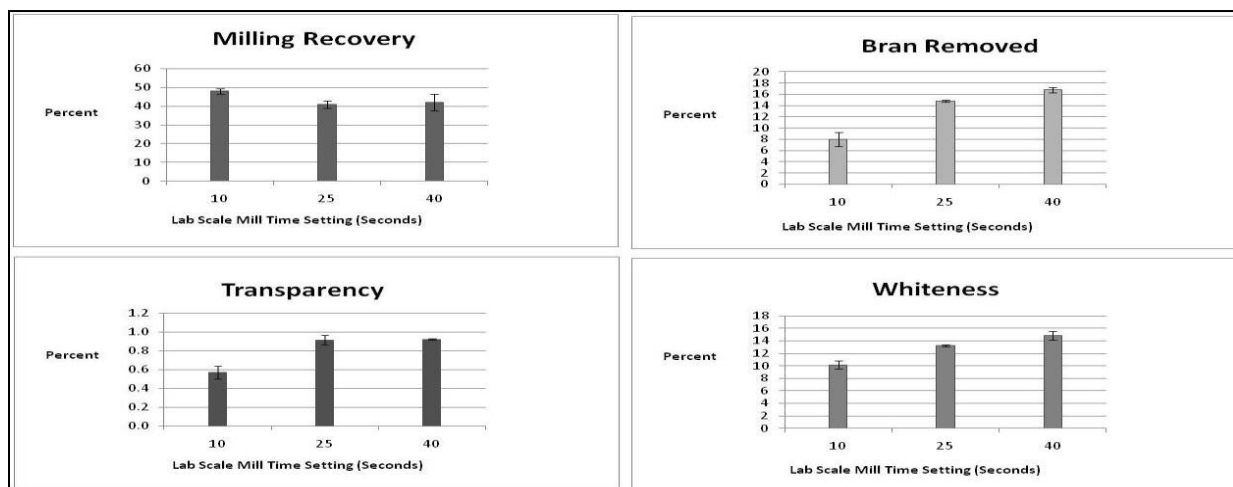
#### 4.2.4 Statistical Analysis

Experimental designs employed a completely randomized factorial format for quality parameters and anthocyanin content for line number MCR02-1576. Three replicates were performed for each time, parameter combination. Evaluation for differences in the mean values of milling quality parameters tested and anthocyanin concentrations measured were conducted at the 5 percent significance level (27). Statistical analysis for anthocyanin concentration and milling quality was performed with ANOVA and Tukey mean comparisons using XLSTAT-Pro (2009) (27).

## 4.3 Results

### 4.3.1 Milling Quality

Milling quality assessment data are presented in Figure 4.2. Milling recovery decreased with increasing milling time, indicating that a longer milling time resulted in a higher level of broken kernels. Milling recovery was less than fifty percent on a rough rice weight basis for all times tested. In a study including red rice varieties, milling recovery values ranged from approximately fifty-one percent to sixty-three percent depending on the variety (5). Our head milling recovery was on the low end of values measured for red rice varieties and nearly 15 percentage points below the non-pigmented parent variety. The non-pigmented parent variety, Cypress, has been considered a good-milling variety by industrial scale mills with a head rice recovery of 63.8 percent (6). Milling recovery is dependent on a number of factors, including geographical, harvest, and milling conditions. These influencing factors could be manipulated to increase milling recovery, but the low milling recovery of this rice line, less than fifty percent, probably precludes further development unless other factors such as value-added processing make further development of the variety economically feasible.



**Figure 4.2. Milling recovery, percent bran removed, transparency, and whiteness; means  $\pm$  one standard deviation indicated**

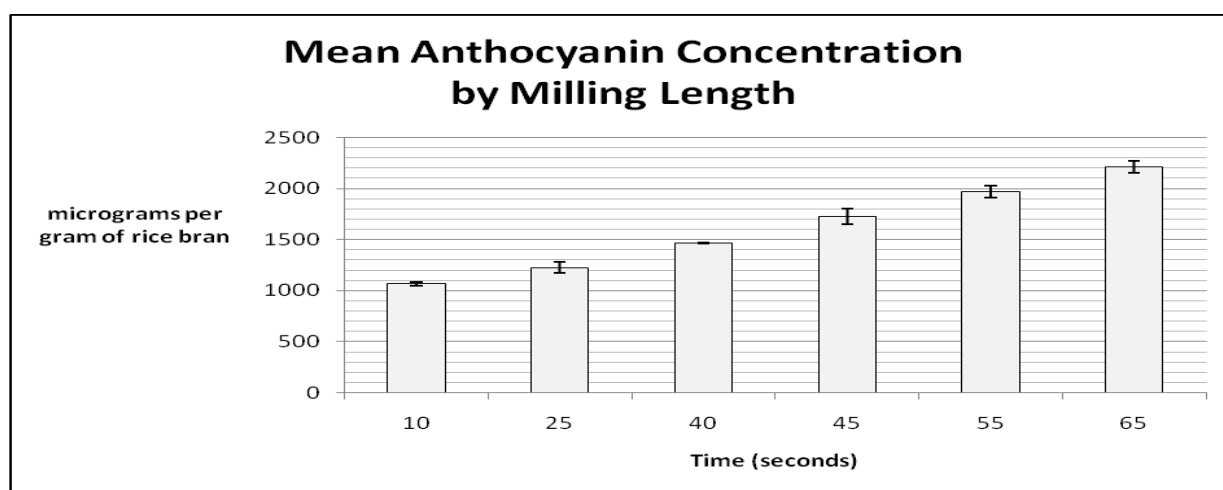
Milling meter measurements of DOM, whiteness, and transparency for line MCR02-1576 provide data to explore the potential for the development of baseline values for purple rice varieties. For the purple rice studied, whiteness values parallel the percent bran removed per milling time, both increasing with longer milling times. The whiteness value was highest at 40 seconds of milling, and ranged from 10.2 to 14.9 percent. Transparency values were not significantly different after the shortest milling time, with values at longer milling lengths near one. DOM values were not measurable for the purple-pigmented rice kernels. Degree of milling, whiteness, and transparency values in non-pigmented varieties exhibit values near 90, between 40 and 60 (3), and between 3 and 4, respectively (4). Visual observation of the milled rice from all mill settings found purple pigment remaining in the kernels. Whiteness and transparency values measured three to four times less than non-pigmented rice varieties, supporting the visual observation (4). The developer of the rice line indicated that for purple pigment to remain in the kernels after milling was unusual (2).

#### **4.3.2 Anthocyanin Concentration**

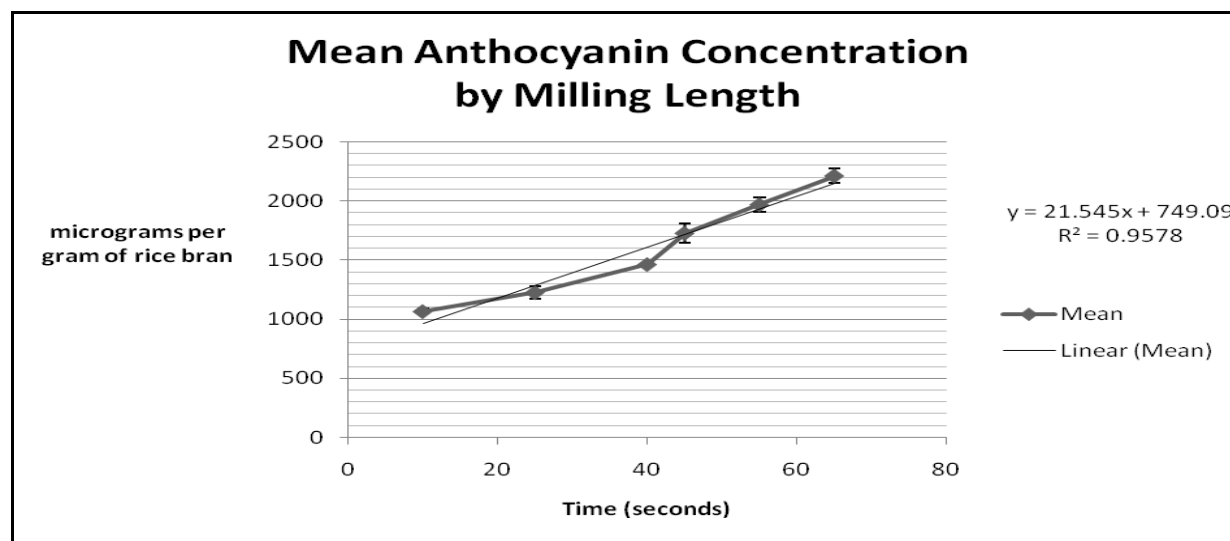
Figure 4.3 shows anthocyanin concentration as a function of milling length. An anthocyanin gradient exists across the bran layer, with anthocyanin concentration increasing with lengthening milling time. Precise fractionation of the bran layer shows a linear relationship existed between anthocyanin concentrations and milling lengths. Figure 4.4 presents the linear relationship, trend line, and equation with coefficient of determination. The results indicate that anthocyanin is concentrated in the inner portion of the bran layer.

The anthocyanin concentration found in this variety is consistent with published values in literature, although these values vary widely. Abdel-Aal et al. (28) determined that anthocyanin concentration in grains varied from 7 to 3276 micrograms of anthocyanin per gram of ground

cereal material. Ichikawa et al. (29) identified that blueberries have 11 anthocyanins while purple rice has predominately one, cyanidin 3-O-beta-D-glucoside (Cy 3-Glc) (29). Nam et al. found four rice varieties in a study of 21 pigmented rice varieties that displayed strong antioxidant activity (22). When extracts from the bran of pigmented rice varieties were compared to extracts from non-pigmented varieties, the extracts from pigmented varieties exhibited higher activity in chemical and mammalian cell studies examining anti-oxidative, anti-tumor, and anti-carcinogenic potential (30).



**Figure 4.3. Anthocyanin concentration; means  $\pm$  one standard deviation**



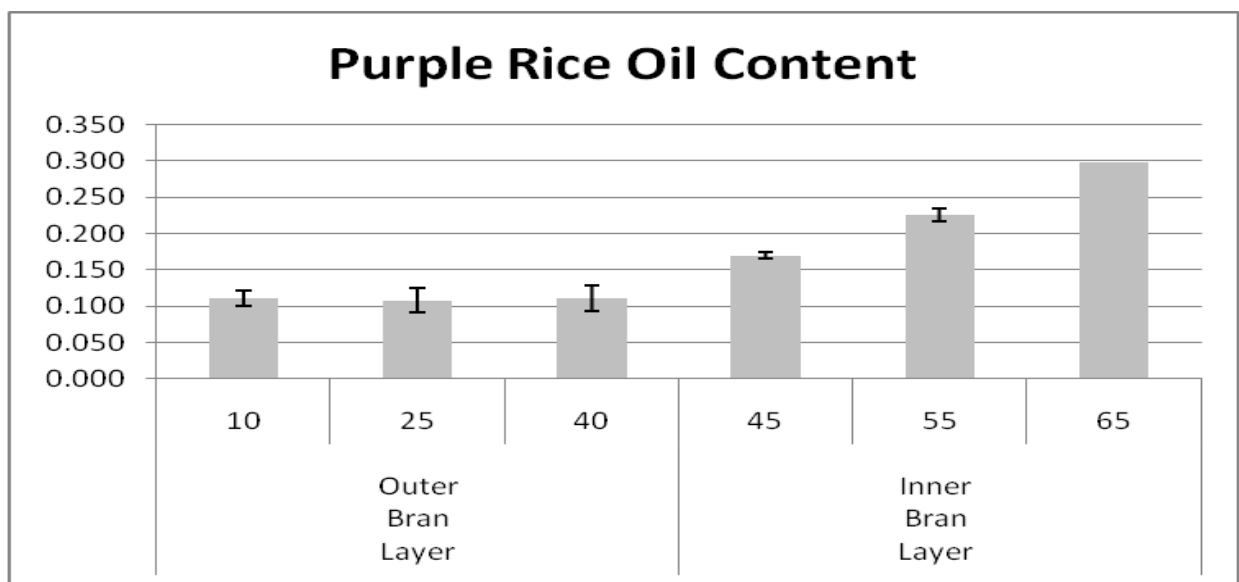
**Figure 4.4. Regression of mean anthocyanin concentration and milling length**

Jang and Xu (23) recently characterized anthocyanin content in a different purple rice variety; these investigators created outer and inner bran layers by milling with a McGill mill at milling length to 40 seconds, and between 40 and 60 seconds. Their findings showed anthocyanin concentration in the inner bran layer to be eight times that of the outer bran layer, with inner and outer concentrations of  $29.0 \pm 0.9$  and  $3.5 \pm 0.4$  mg/g, respectively (23). Dividing our results into two layers by data collected from milling lengths to 40 seconds and after 40 seconds, results in inner and outer bran layers with anthocyanin concentrations of  $2.0 \pm 0.2$  and  $1.3 \pm 0.2$  milligrams per gram of rice bran. The anthocyanin concentration measured in the inner bran layer of our variety was significantly higher (1.6 times) than that measured in the outer bran layer. Literature illustrated the great variability of anthocyanin concentration in grains and fruit sources.

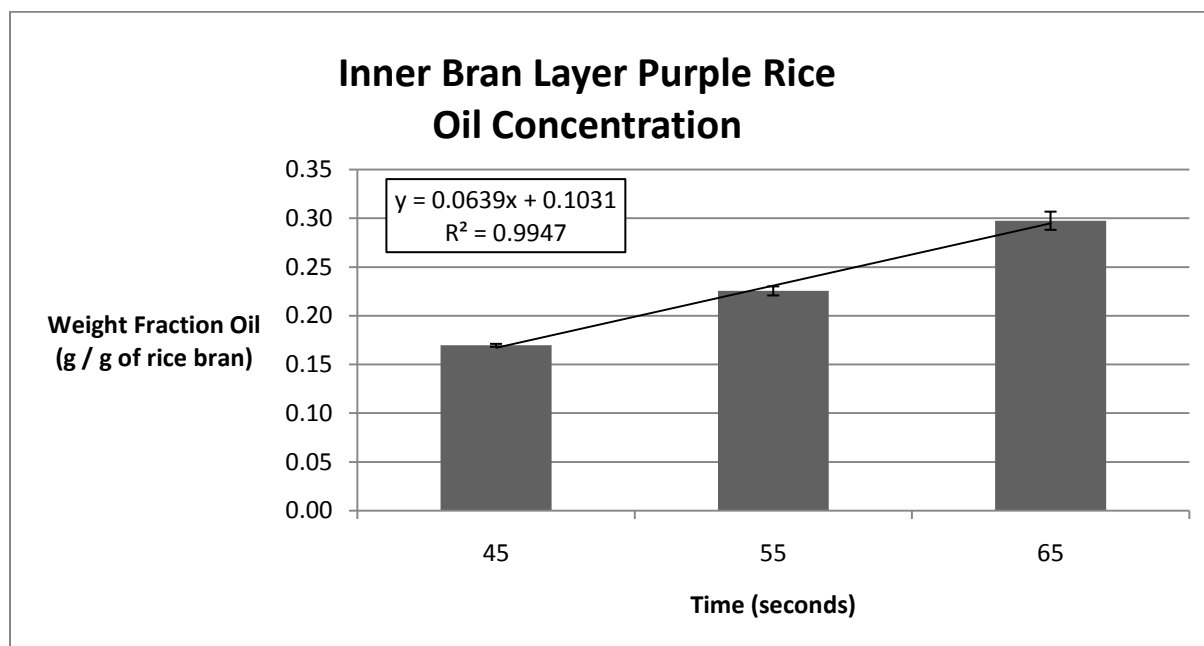
At 1970 micrograms per gram of rice bran, our finding for mean anthocyanin concentration in the inner bran layer was within the range of reported anthocyanin concentrations in the literature. Our inner bran anthocyanin concentration was near the mid-range for cereal (28) and within the range for huckleberries (31). Despite a difference in total anthocyanin concentration, our results followed the same trend as Jang and Xu (23), with the higher anthocyanin concentration located in the inner portion of the bran layer. The more precise fractionation of the rice bran layer employed in our study revealed a linear relationship across the bran layer. Extraction methods employed to obtain anthocyanins should focus on using the inner bran layer as raw material. The differences observed in anthocyanin concentration across the bran layer and between rice varieties indicated a great opportunity to increase anthocyanin concentration through a rice breeding program.

### 4.3.3 Oil Concentration

Figure 4.5 presents oil concentrations across the bran layer and indicates the two divisions of the bran layer. No significant difference for oil concentration existed between successive milling durations for the outer portion of the bran layer. The inner bran layer contained 22 percent oil, approximately twice the mean oil concentration present in the outer bran layer. The inner bran oil concentration compared well with the oil concentration from non-pigmented rice varieties which generally ranges from 18 to 23 percent by weight (7). Figure 4.6 presents data on the inner portion of the bran layer. Oil concentration increased with milling duration. A linear relationship existed for oil concentration across the inner bran layer and milling length. Significant differences existed for oil concentration between successive milling lengths. With rice bran oil, a renewable plant source of oil, currently receiving increased attention as a potential stock material for biodiesel (18; 32), the identification of high oil varieties of rice and identification of the location of the highest concentration of oil in the bran layer would facilitate increased oil recovery through value-added processing.



**Figure 4.5. Oil concentration presented by milling duration with the inner and outer portions of the bran layer indicated; means  $\pm$  one standard deviation**



**Figure 4.6. Oil concentration across the inner bran layer; means  $\pm$  one standard deviation**

## 4.4 Conclusions

The whole kernel milling recovery of line number MCR02-1576 was less than fifty percent, approximately 15 points below Cypress, the non-pigmented parent variety. The inherent purple color of the kernel affected the optical method employed to measure whiteness, transparency, and DOM. Whiteness and transparency values were reported to provide a baseline for pigmented varieties. DOM values were not measurable by milling meter. Mean anthocyanin concentration within the purple rice bran layer was within the ranges seen in published literature for grains and fruits. Anthocyanin concentration displayed a linear relationship with the length of milling. A significantly higher concentration of anthocyanin exists in the inner portion of the rice bran layer. Mean oil concentration was determined to be in the range for non-pigmented rice bran oil concentration values. Oil concentration increased linearly across the inner bran layer. The inner bran layer has approximately two times the oil concentration as the outer bran layer. Potential exists for value-added processing by the collection of the inner portion of the rice



bran layer of this variety for extraction of anthocyanins and oil. Screening purple rice varieties to identify those that possess high anthocyanin content in their bran layers would further optimize value-added processing viability.

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## **CHAPTER 5. PILOT SCALE RICE MILL EVALUATION OF OIL CONCENTRATION ACROSS THE BRAN LAYER OF COCODRIE, CLEARFIELD 161, AND JAZZMAN WITH CORRELATION OF NEAR-INFRARED MEASUREMENT AND HEXANE-EXTRACTION**

### **5.1 Introduction**

With increasing world energy consumption, plant sources of oil have been investigated as alternative energy sources. The United States Department of Energy projects a four percent increase between 2007 and 2030 in the contribution from biomass to total energy supply (1). Rice bran, an agricultural waste product from rice milling, conforms to the United States Department of Energy's definition of biomass, which can include waste from agricultural or forest production, waste from cities or industries, or crops grown for energy use (2).

Rice bran oil, a renewable plant source of oil, has been investigated as stock material for biodiesel (2). Oil extracted from rice bran has been converted to biodiesel by a variety of methods including basic (3) and acidic catalyzation (4), lipase-catalyzation (5), and transesterification by tin compounds (6). One strategy for improving process economics for converting rice bran oil to biodiesel is optimization of the feedstock for this procedure. The identification of high oil concentration varieties of rice and determination of the location of the highest concentration of oil in the bran layer would provide avenues to increase oil recovery using a value-added processing approach. With oil concentration in the rice bran layer characteristically varying from 10 to 23 percent by weight (7), the identification of high oil content rice variety improves the potential for oil recovery by a ratio of 2.3 based on these literature values.

A number of different oil extraction methods have been reported in the literature. Kanitkar et al. (2009) explored microwave-assisted extraction of rice bran oil and its effects on oil yield and oil properties (8). Hu et al. (1996) used hexane to extract vitamin E and oryzanol (9); Lakkakula et al. (2003) used ohmic heating with hexane extraction to study rice bran oil (10). Proctor et al. (1994) determined that a simple, ambient temperature method to measure oil concentration obtained results that are within one percent of more complicated methods (13, 11). These investigators found a ninety-three percent recovery of good quality rice bran oil was achieved with a 10 minute, ambient temperature extraction process (11). In contrast, the extraction method using boiling hexane required 20 minutes of heating to achieve a ninety-two percent recovery, and an additional 40 minutes to obtain an additional one percent recovery (11).

Another method for determining oil concentration in grains employs the use of near infrared transmittance technology, abbreviated NIT (12). Measurement of oil concentration by NIT is an established method for soybeans and corn and has received approval from the United States Department of Agriculture's Grain Inspection, Packers, and Stockyards Association (GIPSA) (12). Rice mills are beginning to use NIT analysis to measure oil concentration because of its rapid measurement, and with calibration, reproducible with limited training of personnel (12).

The identification of high oil content in a variety of rice or the identification of high oil concentration in a portion of the bran layer increases the potential for oil recovery. Three rice varieties, Cocodrie, Clearfield 161, and Jazzman, grown in the southeastern United States were selected for oil concentration evaluation. Cocodrie and Clearfield 161 are established rice varieties in Louisiana. Cocodrie, a long grain high yield variety, was developed and released by the LSU AgCenter in 1998 (13), and accounted for 22.4% of long grain rice acreage planted in

Louisiana in 2008 (14). Clearfield 161, a long grain variety resistant to imidazolinone herbicides, was developed by the LSU AgCenter in conjunction with BASF and released in 2002 (15). Clearfield 161, good-milling, high-yield variety, was planted on 26.3% of Louisiana's 2008 long grain rice acreage (14). Jazzman, a high-yield, good-milling long grain variety, was developed by the LSU AgCenter, and released in 2009 as the rice station's first aromatic jasmine-type rice (16). Jazzman has the potential to compete for a share of the aromatic rice market in the United States (16). The identification of one of the tested varieties with a high oil concentration across the entire bran layer of a rice variety or high oil concentration in a portion of the bran layer would enhance the economic viability of that variety.

Specific study objectives were:

1. To determine total oil concentration in the bran layer of Jazzman, Clearfield 161, and Cocodrie,
2. To determine oil concentration gradient across the bran layer for Jazzman, Clearfield 161, and Cocodrie, and
3. To correlate NIT measurements and hexane extraction concentrations.

## **5.2 Materials and Methods**

### **5.2.1 Sample Preparation**

Jazzman, Clearfield 161, and Cocodrie were supplied by the LSU Agricultural Center (Louisiana Rice Research Station, Crowley, LA), and remained in cold storage (0°C) until testing. Before processing, rice was removed from cold storage and allowed 24 hours to reach ambient temperature (29°C).

### **5.2.2 Processing**

Samples of 11.5 kilograms were processed in triplicate with a pilot scale Satake (Tokyo, Japan) milling plant at operational settings of 3, 5, and 9 for Jazzman, Clearfield 161 and

Cocodrie. After rough rice was husked (Model GPS300A), shelled rice was milled or whitened (Model VAF10AM). Processing at three operational mill settings created divisions across the bran layer. Three divisions allowed for a more detailed analysis of oil concentration across the bran layer. The settings selected (3, 5, and 9) correspond to low, medium, and high flow rates used by Hua et al. (2006) in an optimization study of Cocodrie (13).

### **5.2.3 Measurements**

Shelled rice, milled rice, and bran samples were collected from tested settings. The amount of bran removed for each mill setting was determined from the difference between the weights of shelled and milled rice, and was reported as a weight percentage of bran removed to shelled rice processed. Bran oil concentration was determined as a weight fraction of oil extracted with hexane to the amount of bran processed. Milled rice oil concentration was measured with NIT.

#### **5.2.3.1 Hexane Extraction**

For bran samples collected at pilot scale mill settings of 3, 5, and 9, rice bran oil was extracted using the method detailed in Proctor et al. (11). A brief description of this method includes the following steps: one gram of rice bran was combined with 10 milliliters of hexane and stirred for a period of 10 minutes. Samples were centrifuged and decanted, and then the solvent was evaporated from the extracted oil. The weight of the oil was measured, and results were reported as a weight fraction of oil recovered to bran processed.

#### **5.2.3.2 Near Infrared Transmittance (NIT) Analysis**

The oil concentration of milled rice samples from pilot scale settings of 3, 5, and 9 were measured with an Infratec™ 1241 Grain Analyzer (Foss Analytical, Denmark) (12). The NIT analyzer separated the sample into 10 sub-samples, measured transmittance, and reported the

mean transmittance value (12). Because NIT analysis is an indirect measurement technique, NIT results must be correlated to measured values. Calibration between transmittance measurements and an established evaluation method can be approached by using a least squares regression (12). Regression models were developed to predict bran oil concentrations from milled rice NIT measurements.

#### 5.2.4 Statistical Analysis

Table 5.1 presents the experimental design. Triplicate measurements were made for near infrared analysis and hexane extractions of oil from rice bran samples. Statistical analysis using Tukey mean comparisons were conducted with XLSTAT-Pro (2009) (17) at a 5 percent significance level.

**Table 5.1. Experiment design, dashes indicate replicates**

Pilot Scale Mill Setting	Oil Concentration	
	Hexane Extraction of Oil from Sample of Bran Removed at Mill Setting	NIT Measurement of Oil Concentration in Milled Rice Collected from Mill Setting
3	— — —	— — —
5	— — —	— — —
9	— — —	— — —

### 5.3 Results

#### 5.3.1 Oil Concentration by Variety

Mean hexane extraction measurements are presented as a weight fraction of oil obtained to bran processed. Statistical analysis of hexane extraction results found Clearfield 161's oil concentration to be higher than Cocodrie or Jazzman. Hexane extraction results in ascending order were Cocodrie, Jazzman, and Clearfield 161. Hexane results ranged from 0.15 to 0.32 weight fraction of oil; Clearfield 161 contained 2.11 times the oil concentration found in

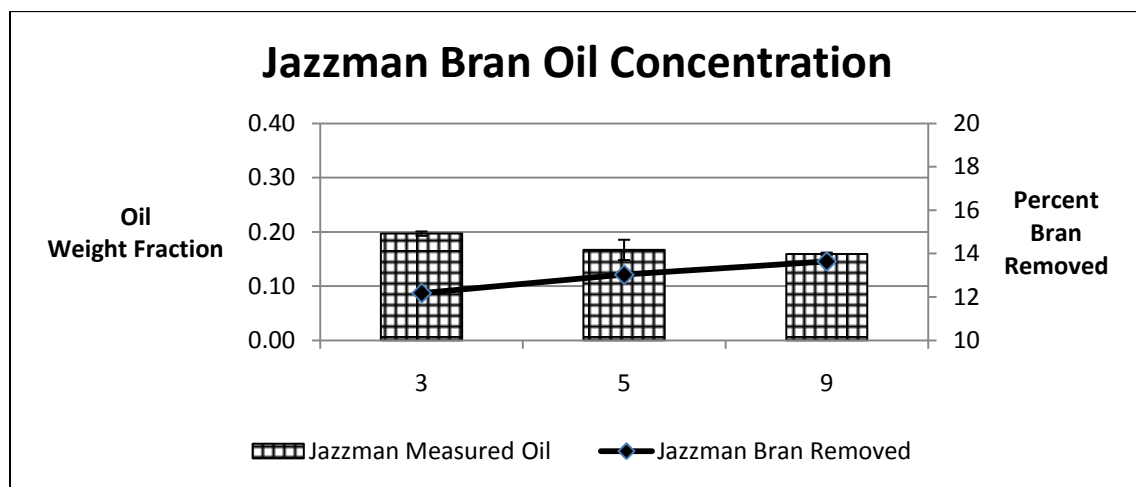


Cocodrie and 1.83 times Jazzman's oil concentration. In previous pilot scale research, the total oil concentration measured by hexane extraction for Cocodrie was  $0.186 \pm 0.008$  as a weight fraction (18) of oil obtained to rice bran extracted. Our current study found a concentration ratio of  $0.151 \pm 0.018$ . This difference was possibly due to variation from different geographic planting locations or from the use of different hexane based extraction methods. The earlier study employed a heated-hexane method with two extractions (18), while this study used a rapid, 10 minute, ambient temperature method with one extraction. The rapid single extraction method typically achieved a 93 percent recovery after 10 minutes, and a boiling single extraction method achieved a 93 percent recovery after 80 minutes (11). Using a 93% recovery and two extractions per bran sample, an oil recovery of 99.5 % could be predicted for the two extraction method. At 99.5 % recovery, the 0.186 weight fraction of oil recovered from two extractions represented a total oil weight fraction of 0.187. For the current study with a 93% oil recovery, the total oil weight fraction was 0.162. Our current study results were 87 percent of the previous two solvent study results using less solvent and time. The oil concentration of Clearfield 161 at a weight fraction of 0.318 is higher than values reported in the literature typically varying between 0.10 and 0.23 (7). Thus, this variety has great potential as a feedstock for biodiesel production.

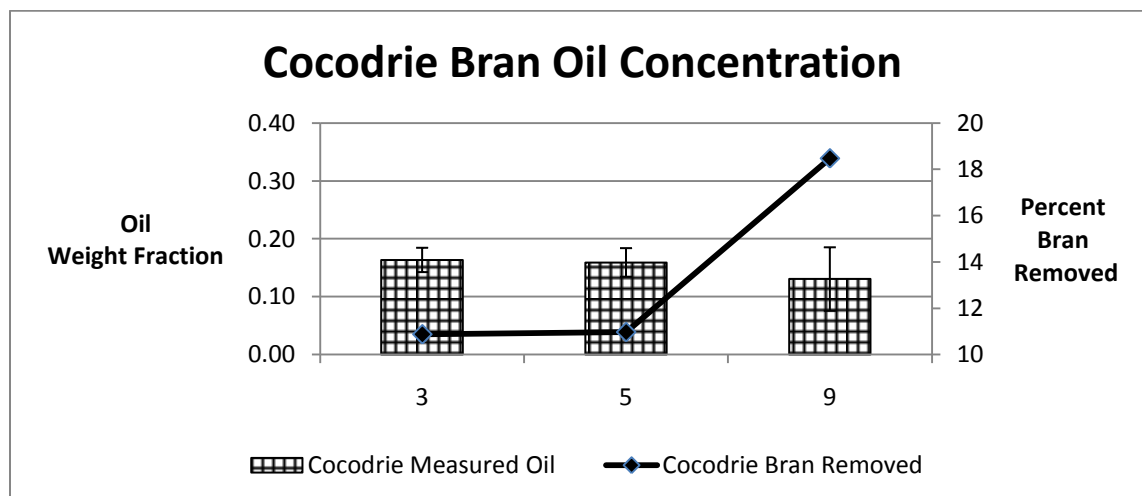
### **5.3.2 Oil Concentration by Bran Division**

Figures 5.1, 5.2, and 5.3 present oil concentrations by rice variety and by pilot scale mill setting. Bran oil concentrations determined by HPLC after hexane extraction were graphed with corresponding values for the weight percentage of bran removed for tested pilot scale settings. The results for Jazzman, Cocodrie, and Clearfield 161 were presented with one standard deviation indicated.

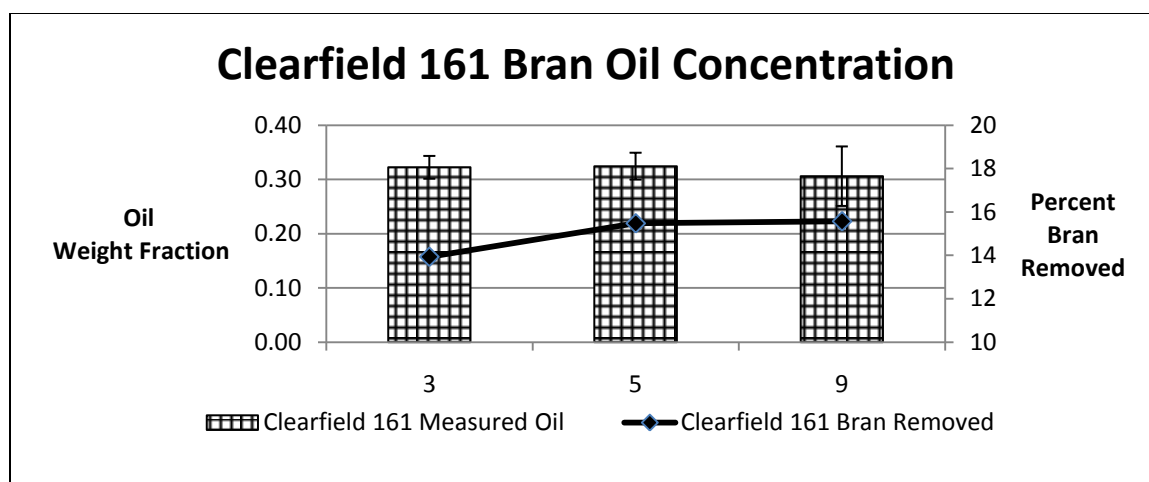
Oil concentration measured by hexane extraction decreased with increasing pilot scale mill setting. Between operational mill settings, Clearfield 161 and Cocodrie exhibited no statistical differences for oil concentration determined by hexane extraction; however, Jazzman exhibited a difference in oil concentration between mill setting 3 and the two remaining mill settings. Our current oil concentration results found decreasing oil concentration with increasing pilot scale mill setting. This is consistent with previous research results for Cocodrie, Cypress, and Cheniere (18).



**Figure 5.1. Jazzman bran oil concentration with  $\pm$  one standard deviation**



**Figure 5.2. Cocodrie bran oil concentration with  $\pm$  one standard deviation**



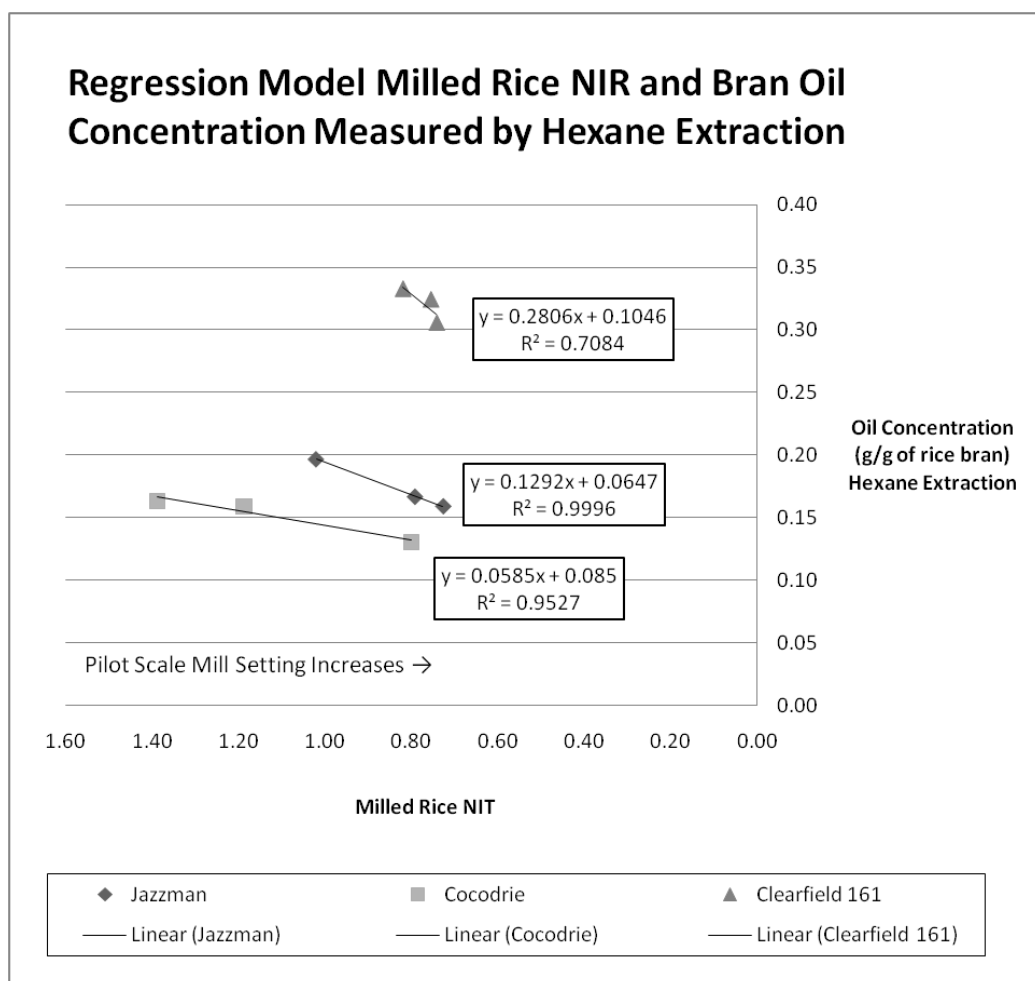
**Figure 5.3. Clearfield bran oil concentration with  $\pm$  one standard deviation**

### 5.3.3 Regression Models for Milled Rice NIT Values and Measured Bran Oil Concentration

From each run of the pilot scale mill, samples of milled rice and bran were collected. NIT measurements were made for the milled rice sample collected; and the oil concentration of the associated bran material determined. To predict bran oil concentration from milled rice NIT measurements, a least squares regression model was developed for tested rice varieties. Figure 5.4 presents the regression models formulated between NIT milled rice values and measured bran oil concentrations.

Table 5.2 presents NIT measurements for milled rice samples collected at pilot scale settings of 3, 5, and 9 and the associated predicted rice bran oil concentrations. Predicted oil values were determined from milled rice NIR and the regression model developed for each rice variety. Predicted oil concentrations trended lower with increasing pilot scale mill setting for tested rice varieties.

Milled rice NIT values were used to predict bran oil concentrations which were compared to measured oil concentrations. Figure 5.5, Figure 5.6, and Figure 5.7 present by variety and pilot scale mill setting the differences between mean measured and mean predicted bran oil concentrations. Predicted oil concentrations were within two percent of the measured values.

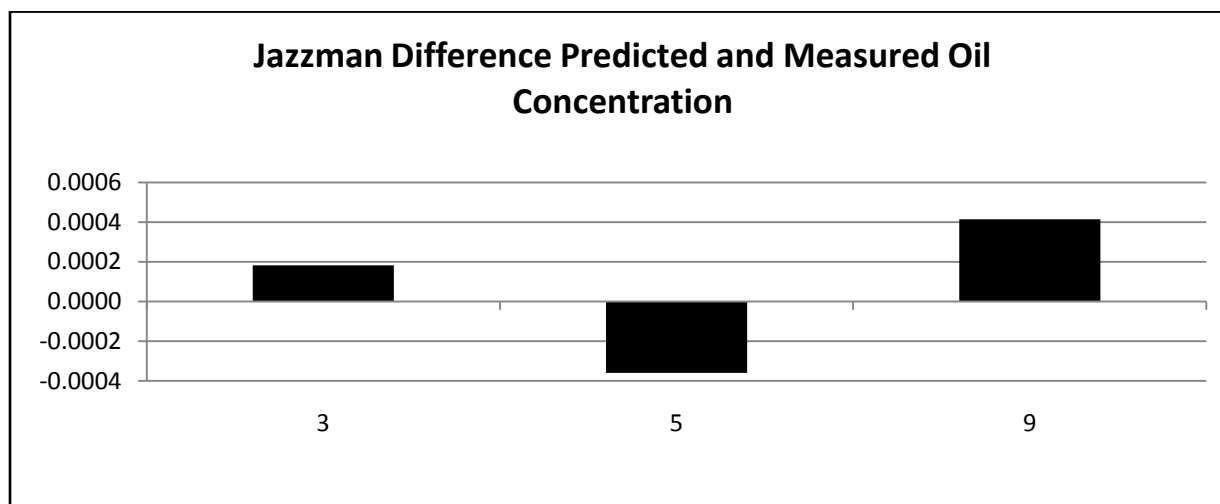


**Figure 5.4. Regression models milled rice NIR and measured bran oil concentration**

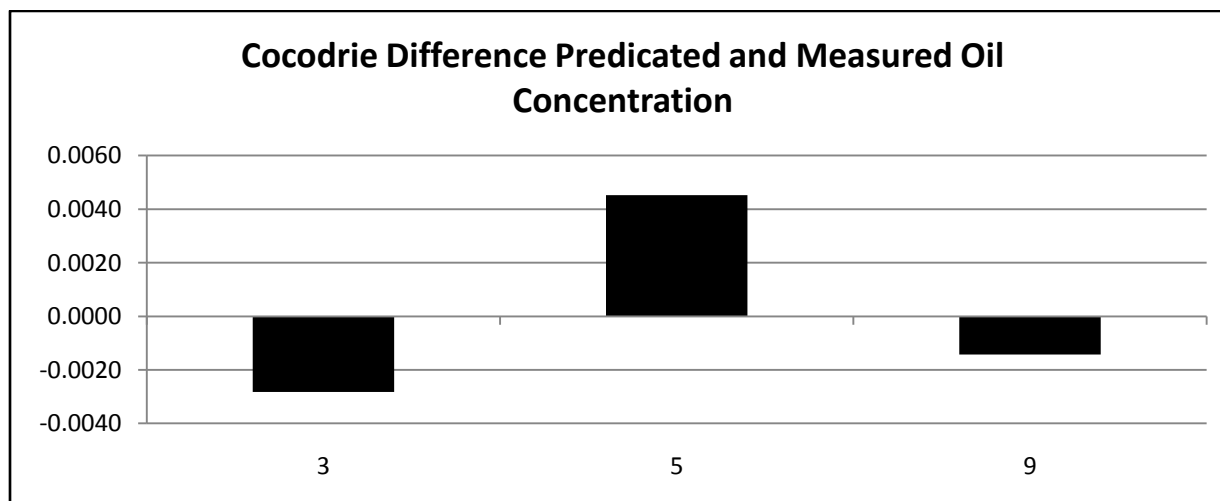
**Table 5.2. Jazzman, Cocodrie, and Clearfield 161 milled rice NIT measurements and associated bran oil concentration**

Rice Variety	Pilot Scale Setting	NIT	Oil Concentration Bran	Predicted Oil
Jazzman	3	1.020 ± 0.19	0.197 ± 0.00	0.196 ± 0.02
	5	0.792 ± 0.06	0.167 ± 0.02	0.167 ± 0.01
	9	0.727 ± 0.10	0.159 ± 0.00	0.159 ± 0.01
Cocodrie	3	1.387 ± 0.11	0.163 ± 0.01	0.166 ± 0.01
	5	1.188 ± 0.37	0.159 ± 0.04	0.154 ± 0.02
	9	0.799 ± 0.13	0.130 ± 0.03	0.132 ± 0.01
Clearfield 161	3	0.819 ± 0.02	0.333 ± 0.01	0.334 ± 0.01
	5	0.755 ± 0.00	0.324 ± 0.02	0.316 ± 0.00
	9	0.742 ± 0.01	0.306 ± 0.01	0.313 ± 0.00

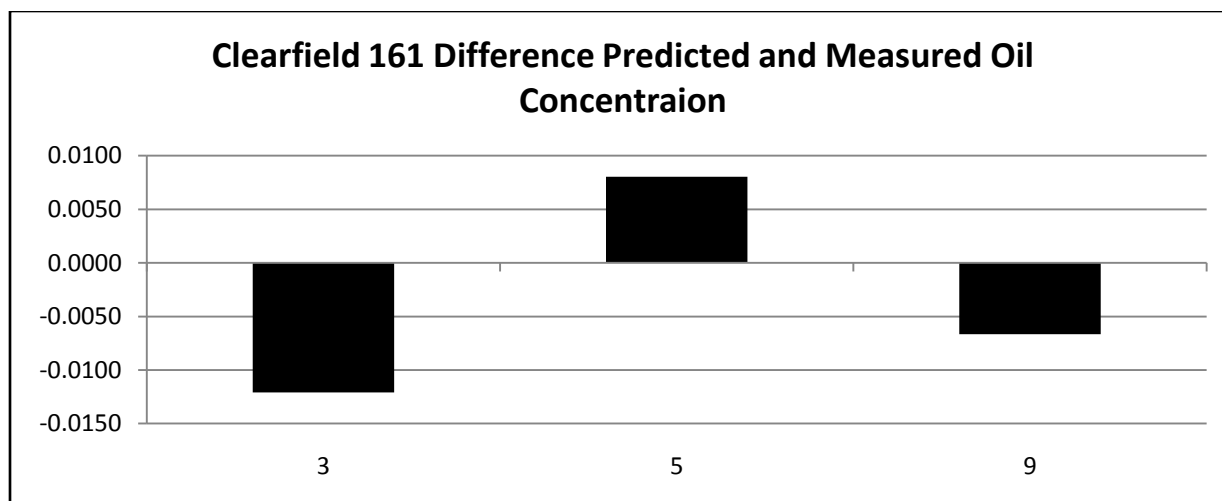
Predicted bran oil concentrations overestimate for pilot scale setting 5 and underestimate for pilot scale setting 3 for Cocodrie and Clearfield 161. The regression model for Jazzman predicts oil concentrations that over estimate for pilot scale mill settings of 3 and 9, while underestimating for setting 5. Tukey mean comparisons were conducted between predicted and measured oil concentration values. No statistical differences between measured and predicted bran oil concentrations existed at any pilot scale setting for tested rice varieties.



**Figure 5.5. Differences in oil concentration predicted by regression model and measured by hexane extraction for Jazzman**



**Figure 5.6. Differences in oil concentration predicted by regression model and measured by hexane extraction for Cocodrie**



**Figure 5.7. Differences in oil concentration predicted by regression model and measured by hexane extraction for Clearfield 161**

## 5.4 Conclusions

Hexane extraction results found that Clearfield 161 had significantly higher total oil content than Jazzman or Cocodrie, 1.83 and 2.11 times, respectively. Within variety, Clearfield 161 and Cocodrie showed no difference in oil concentration across the bran layer for hexane extraction. Milled rice NIT measurements and regression models were used to predict the oil concentration within the bran layer. Regression models were developed for each rice variety tested. Predicted oil concentrations were within two percent of the measured values. Predicted bran oil concentrations overestimate for pilot scale setting 5 and underestimate for pilot scale setting 3 for Cocodrie and Clearfield 161. The regression model for Jazzman predicts oil concentrations that over estimate for pilot scale mill settings of 3 and 9, while underestimating for setting 5. A predictive model provides information useful to rice farmers who produce rice and to rice millers who process rice to commercial expectation or specified customer request. Rice varieties, with high oil content in their bran, have potential as a renewable, plant source for oil, and are usable in nutritional, pharmaceutical, or industrial applications.

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## **CHAPTER 6. CONCLUSIONS AND RECOMMENDATIONS FOR ADDITIONAL RESEARCH**

Value-added processing of rice and rice bran is important to optimize milled rice quality and to increase the recovery of oil and phytochemicals from the bran layer. Optimizing the milling process increases milling yields resulting in higher economic return. Processing the bran layer into divisions permits the use of separate fractions for recovery of high-value components of interest. Recovery of high-value components while reducing the total amount of bran requiring processing impacts the economics of the extraction process by reducing material handling and processing costs.

### **6.1 Conclusions: Scientific**

Clearfield 161 was milled at laboratory, pilot, and industrial scales. For each mill scale, rice quality was determined for Clearfield 161. Predictive comparisons for milling quality among the three different mill scales were identified. Improved correlation of rice quality parameters across the mill scales increases the information available for process optimization, thus impacting milling yield and economic return for this and potentially other rice varieties being developed.

Aromatic and purple rice varieties fill a niche in the rice specialty market. Information about these new varieties is important because they have the potential to expand the domestic market for specialty rice by capturing a part of the import market to the United States. Pilot scale evaluation of Jazzman, a recently released aromatic, long-grain rice variety, characterized this new variety while providing information for optimization of the milling process. Jazzman presented as a high yield, good milling aromatic rice variety, with yields and milling quality comparable to two commercially successful non-aromatic varieties grown in the same area and

to two long grain aromatic varieties grown in Bangladesh. Jazzman has the potential to compete for a share of the aromatic rice market in the United States.

A purple rice variety, line number MCR02-1576, was milled to characterize milling quality and to evaluate anthocyanin and oil concentrations across the bran layer. The whole kernel milling recovery was less than fifty percent, and the inherent purple color of the kernel affected the optical method employed to measure whiteness, transparency, and DOM. Mean anthocyanin concentration within the purple rice bran layer was within the ranges seen in published literature for grains. A significantly higher concentration of anthocyanin exists in the inner portion of the rice bran layer. Mean oil concentration was determined to be in the range for non-pigmented rice bran oil concentration values. The inner bran layer has approximately two times the oil concentration as the outer bran layer. Potential exists for value-added processing by the collection of the inner portion of the rice bran layer of this variety for extraction of anthocyanins and oil. Screening purple rice varieties to identify those with high anthocyanin content increases the economic viability of that variety for use in a value-added processing approach.

Pilot scale milling of Jazzman, Clearfield 161 and Cocodrie provided samples of bran for determination of bran oil concentration and of milled rice for NIT measurements. Hexane extraction results found that Clearfield 161 had significantly higher total oil content than Jazzman or Cocodrie, 1.83 and 2.11 times, respectively. Within variety, Clearfield 161 and Cocodrie showed no difference in oil concentration across the bran layer for hexane extraction. Milled rice NIT measurements and regression models were used to predict the oil concentration within the bran layer. Regression models were developed for each rice variety tested. Predicted oil concentrations were within two percent of the measured values. Predicted bran oil

concentrations overestimate for pilot scale setting 5 and underestimate for pilot scale setting 3 for Cocodrie and Clearfield 161. The regression model for Jazzman predicts oil concentrations that over estimate for pilot scale mill settings of 3 and 9, while underestimating for setting 5. A predictive model provides information useful to rice farmers who produce rice and to rice millers who process rice to commercial expectation or specified customer request. Rice varieties with high oil content in their bran have potential as a renewable, plant source for oil, and are usable in nutritional, pharmaceutical, or industrial applications.

## **6.2 Conclusions: Economic Impact**

### **6.2.1 Broken Kernels**

Reducing the percentage of broken kernels increases the economic performance of a rice variety. The benchmark for the global rice market is the price for *Thai 100% B 2<sup>nd</sup> Grade* which had a mean value of 587 U.S. dollars per metric ton (metric ton) for 2009 (1). The mean value for rice with broken kernels at five percent by weight was 555 U.S. dollars per metric ton for 2009 (1). Using global market prices per metric ton and the U.S. long grain rice production for 2009 as a basis for illustrating economic impact, a nearly 38 million dollar loss would have occurred for long grain U.S. rice production in 2009 with an increase in broken kernels of only five percent by weight (1, 2). At 25 percent broken kernels, the price per metric ton was 127 U.S. dollars per metric ton which would have represented an 836 million U.S. dollar decrease in value from the benchmark. These examples illustrate that optimization of the milling process to reduce the occurrence of broken rice kernels could have a positive economic impact.

### **6.2.2 Specialty Varieties**

Specialty varieties bring a higher price per metric ton on the global market. *Thai fragrant (100%)* sold for 954 U.S. dollars per metric ton and *Pak Basmati Ordinary* sold for 937 U.S.

dollars per metric ton in 2009 (1). With *Thai 100% B 2<sup>nd</sup> Grade* selling for 587 U.S. dollars per metric ton, the value of aromatic rice exceeded this benchmark for non-aromatic rice by over 300 U.S. dollars per metric ton in 2009 (1). The newly released rice variety, Jazzman, has the potential to replace a portion of the approximately 350 thousand metric tons of aromatic rice imported to the United States (1). If Jazzman captured a third of the aromatic rice import market, it would represent an increase in value of 35 million U.S. dollars over non-aromatic rice at 2009 per metric ton rice prices (1).

### **6.2.3 Rice Bran Oil**

Biodiesel refineries recently built in the southern United States range in capacity from 151 to 397 liters per year (3). An estimated 87 million liters of biodiesel could be produced from the amount of rice bran milled from the U.S. long grain rice production of 2009. This production estimate was based on rice bran containing 20% oil by weight, a 90% recovery of oil by solvent extraction, a 60% conversion of rice bran oil to biodiesel, and a mean biodiesel density of 0.88 g/ml (4). Eighty-seven million liters indicates that rice bran could provide approximately 20% of the yearly feed stock requirement for the larger refinery and over half for the smaller refinery. Approximately thirty-three million liters of biodiesel could be produced from the amount of bran removed from Louisiana's 2009 long grain rice production which represented 8 and 19 percent of the feed stock requirements for the two refineries (5).

## **6.3 Recommendations**

Value-added processing of rice and rice bran positively impacted economic worth. Decreasing broken kernels and optimizing milling for new varieties increased the value of milled rice produced. Processing the bran layer into divisions permitted the use of separate fractions for

recovery of high-value components impacting the economics of the extraction process by reducing material handling and processing costs. This research suggested future research areas. To enhance predication of milling performance, different types of rice including short grain, medium grain, aromatic varieties, and additional long grain varieties could be milled at laboratory, pilot, and industrial scale. Since Jazzman is a new aromatic rice variety release, future bran studies might characterize the aromatic compounds present by identifying, quantifying, and assessing their concentration across the bran layer. DOM and whiteness values possess potential as predictive measures for acceptability of aroma and final product quality for aromatic rice varieties. Future work with the purple rice line studied could focus on increasing milling recovery and investigating the purple pigment remaining in the kernel to explore if the remaining anthocyanin concentration reaches levels viable for health benefits. Further testing with hexane extractions and NIT measurements could lead to better predictive models for determining bran oil concentration.

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## APPENDIX A: MULTI-SCALE DATA FOR CLEARFIELD 161

Clearfield 161 for Laboratory, Pilot, and Industrial Scale						
Mill Scale	Operational Control Setting	Milling Yield (%)	Bran Removed (%)	Whiteness	Transparency	Degree Of Milling
Lab	10	91.0	7.5	59.9	2.8	185
Lab	10	90.7	7.9	59.4	2.55	181
Lab	10	90.5	7.9	59.8	2.77	184
Lab	25	94.7	15.5	60.8	2.58	188
Lab	25	91.3	10.4	69.1	3.27	199
Lab	25	91.5	12.0	70.2	3.32	199
Lab	40	92.7	16.5	74.9	3.69	199
Lab	40	92.7	14.4	73.4	3.47	199
Lab	40	94.0	13.8	72	3.82	199
Pilot	3	92.5	14.2	69.2	3.44	199
Pilot	3	94.9	13.3	71.1	3.33	199
Pilot	3	89.6	14.2	56	2.41	164
Pilot	5	92.0	14.5	70.8	3.58	199
Pilot	5	89.1	17.6	71	3.65	199
Pilot	5	89.9	14.3	72.6	3.35	199
Pilot	9	89.1	15.4	71.6	3.35	199
Pilot	9	90.1	16.6	70.9	3.15	199
Pilot	9	89.3	14.7	72.8	3.69	199
Industrial	Break 1	87.4		58.1	0.84	171
Industrial	Break 1	85.4		57	0.89	166
Industrial	Break 1	84.8		56.4	0.62	163
Industrial	Break 2	87.6		50.2	1.10	180
Industrial	Break 2	88.1		59.3	1.16	176
Industrial	Break 2	87.2		60.2	0.90	181
Industrial	Break 3	87.3		69.4	1.68	199
Industrial	Break 3	88.5		65.7	1.61	199
Industrial	Break 3	89.7		65.7	1.65	199

## APPENDIX B: PILOT SCALE DATA FOR JAZZMAN

Pilot Scale Mill Data Collection Sheet																		
Variety: New Jasmine Variety																		
Run	Tare weight (kg)	Mill Setting	Milled Rice Flow Rate (kgs / sec)			Flow Rate (kgs/sec)	Flow Rate (kgs/hr)	Shelled Rice					Milled Rice					Bran
			Time (sec)	Weight with container	Weight (kgs)			(kgs) w/bucket	kgs	DOM	W	T	(kgs) w/bucket	kgs	DOM	W	T	(kgs)
7	0.635	3	7.01	0.315	0.270	0.039	139	13.185	12.550	0	20.7	0.87	11.890	11.255	49	29.7	2.74	1.295
3	0.635	3	9.08	0.320	0.275	0.030	109	11.525	10.890				9.970	9.335	76	34.1	3.82	1.555
5	0.740	3	7.75	0.310	0.265	0.034	123	12.075	11.335				10.720	9.980	69	32.9	3.55	1.355
9	0.605	5	2.94	0.315	0.270	0.092	331	12.580	11.975				11.285	10.680	83	36.3	3.43	1.295
1	0.740	5	3.21	0.360	0.315	0.098	353	11.420	10.680				9.650	8.910	77	34.9	3.46	1.770
4	0.625	5	3.25	0.360	0.315	0.097	349	11.700	11.075				10.405	9.780	80	35.5	3.58	1.295
8	0.630	9	1.31	0.345	0.300	0.229	824	11.585	10.955				10.280	9.650	87	37.2	3.36	1.305
6	0.605	9	1.35	0.340	0.295	0.219	787	13.195	12.590				11.675	11.070	88	37.3	3.43	1.520
2	0.740	9	2.24	0.390	0.345	0.154	554	11.135	10.395				9.375	8.635	84	36.5	3.53	1.760

## APPENDIX C: ANTHOCYANIN CONCENTRATION DATA BY HPLC PEAK

Data Collection Sheet											
Run		Anthocyanin Concentration									
		Peak 1			Peak 2			Peak 3			Total
	Time Setting	area	ug of a. in 25 µl	ug of a. in 3 ml or in 0.5 gram rice bran	area	ug of a. in 25 µl	ug of a. in 3 ml or in 0.50 gram rice bran	area	ug of a. in 25 µl	ug of a. in 3 ml or in 0.5 gram rice bran	ug of a. in 3 ml or in 1 gram rice bran
1	10	2060320	0.343	41.206	21957695	3.660	439.154	2284076	0.381	45.682	1052.084
4	10	1937617	0.323	38.752	19265878	3.211	385.318	2298148	0.383	45.963	940.066
6	10	2206224	0.368	44.124	22487315	3.748	449.746	2318652	0.386	46.373	1080.488
2	25	3048759	0.508	60.975	30074219	5.012	601.484	3309418	0.552	66.188	1457.296
5	25	2498107	0.416	49.962	24566951	4.094	491.339	2656134	0.443	53.123	1188.848
7	25	1884406	0.314	37.688	26918170	4.486	538.363	2807552	0.468	56.151	1264.405
3	40	2986683	0.498	59.734	30347953	5.058	606.959	3218983	0.536	64.380	1462.145
8	40	3433872	0.572	68.677	30036977	5.006	600.740	3165211	0.528	63.304	1465.442
9	40	2803399	0.467	56.068	25734487	4.289	514.690	2787913	0.465	55.758	1253.032

## APPENDIX D: NEAR INFARED MEASUREMENTS BY VARIETY

NIT Measurement Milled Rice			
Pilot Scale Setting	Jazzman	Cocodrie	Clearfield 161
3	1.216	1.275	0.805
3	0.833	1.384	0.972
3	1.011	1.503	0.833
5	0.849	1.320	0.752
5	0.722	0.770	0.758
5	0.805	1.473	0.744
9	0.804	0.859	0.752
9	0.759	0.885	0.731
9	0.617	0.654	0.706

## **VITA**

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