<u>Progress Report_1st Month</u>

Development of test method(s) for determination of degree of milling of rice (FAD 0218)

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Project Investigator(s)

Dr. Brajesh Kumar Panda **Dr. Gayatri Mishra** **Dr. Ronit Mandal**

Agricultural and Food Engineering Department

Indian Institute of Technology Kharagpur

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1. Project Overview

Background and Rationale: The degree of milling directly affects rice quality, impacting nutritional value, cooking characteristics, and shelf life. Current methods for determining DOM often vary by region and cultivar, resulting in inconsistencies in quality assessments. This project seeks to address these inconsistencies by developing a method that can reliably quantify DOM across all commercial rice cultivars and be adopted as an Indian standard, with potential for international alignment.

Objective: To develop test method(s) for determination of degree of milling of rice suitable for commercial cultivars and to validate the protocol(s) for uniformity and robustness**.**

2. Review of Literature

A thorough literature review has been conducted for the project, covering existing methods for determining the degree of milling (DOM), associated chemical changes in rice during milling, and imaging techniques used to assess DOM and other quality attributes. This review includes analyses of physical and chemical testing methods traditionally used for bran quantification, alongside advancements in machine vision for non-destructive measurement of surface characteristics, which capture essential details like kernel shape, size, and texture. This foundation guides the development of a comprehensive, standardized approach to accurately measure DOM across rice varieties.

Chen et al. (2022) developed an advanced image-based deep learning model to classify the degree of milling (DOM) in rice. Using the Inception ResNet–Bayesian Optimization Algorithm (IRBOA), this model classified rice as well-milled, reasonably well-milled, or substandard with high accuracy. Image data of rice samples were collected using a Phantom h9 flatbed scanner, and with a dataset of 17,400 images, the model achieved an average accuracy of 96.90% across categories. This study highlights the effectiveness of combining image processing with deep learning to improve DOM classification for rice quality control.

Kale et al. (2019) analyzed mineral content in raw and parboiled PB1121 rice across various degrees of milling (DOM) using di-acid digestion and advanced colorimetric and spectrophotometric methods, including a Spectrophotometer (for P, S), Flame Photometer (for K), and Atomic Absorption Spectrophotometer (for other minerals). The study found that P, Mg, and Fe decreased as DOM increased in both rice types, indicating these minerals are primarily in the bran and outer layers, while

Mn, Zn, Cu, and S remained stable across DOMs, suggesting uniform distribution. K, P, and Fe levels were consistently higher in parboiled rice, indicating mineral retention during parboiling. Total mineral content showed that raw rice had higher minerals at lower DOM (0% and 5%), while parboiled rice retained more minerals at DOM ≥ 6%. Findings suggest parboiling enhances mineral retention in polished rice, adding nutritional value post-milling.

Lamberts et al. (2007) evaluated the effects of milling on rice's color and nutritional profile, particularly protein and mineral content. Using an automated Dumas protein analysis system (EAS vario Max N/CN) and AOAC/AACC methods, they analyzed protein (conversion factor 5.95 from nitrogen) and minerals. Results showed a decline in protein from 9.2% at 0% DOM (brown rice), with proteins concentrated mainly in the bran (15.8%) and outer endosperm. Mineral content also decreased with DOM, stabilizing beyond 15% DOM. With 61.0% of minerals in the bran versus 11.6% in the core endosperm, this study reinforced that bran and outer endosperm layers are key for nutrient retention, supporting targeted milling practices to preserve rice nutrition.

Mardison et al. (2018) developed a mathematical model for determining the degree of milling (DM) of rice by measuring UV absorbance at 331 nm. Using the equation

$$\text{DM model} = 12.613 \times e^{-0.987 \times A_{331}}$$

Where, A = UV absorbance value at wavelength 331 nm

The study showed that DM could be estimated by dissolving 10 g of rice in 20 ml of n-hexane and measuring absorbance after 2-3 hours. While promising, further validation is required to confirm this model’s accuracy.

Miller et al. (1978) introduced a rapid and objective method to assess the degree of milling (DOM) in rice using electrical conductivity measurements. In this method, 10 g of milled rice is extracted with 40 ml of isopropyl alcohol-water (1:1) for 5 minutes, and the conductivity of the extract is measured at 25°C. The conductivity values correlate with DOM due to differences in ash concentration between the aleurone layer and endosperm. Conductivity values are classified from undermilled to well-milled, providing a practical approach for routine DOM testing.

Paul et al. (2019) investigated the impact of DOM on zinc content in rice, with samples milled to 7.5-15% DOM. Using nitric and perchloric acid digestion and atomic absorption spectrophotometry (AA-7000), they observed a significant reduction in zinc as DOM increased, with a notable decrease up to

12% DOM. Reducing DOM from the commercial standard of 10% to 4-6% could increase zinc content by 27-47%. Although brown rice retains the highest zinc, under-milled rice showed greater consumer acceptance, suggesting that lower DOM rice may offer a viable option for enhancing nutritional content while meeting consumer preferences.

Wan and Long (2010) developed a machine vision method for assessing the degree of milling (DOM) in rice by analyzing surface texture through a gray-gradient co-occurrence matrix. Samples were milled at 30s, 60s, and 90s, producing different DOM levels and compared against a commercial sample. Using a V-groove conveyor, MOTIC stereomicroscope, and SONY camera, the study achieved an average detection accuracy of 94% across all samples. This study demonstrates the effectiveness of image analysis for DOM assessment, highlighting its potential for both research and industrial quality control.

Yadav & Jindal (2001) assessed milling quality in rice by analyzing the degree of milling (DOM) and head rice yield (HRY) across ten varieties, including Suphan Buri and Mali scented rice. Using an imaging system with a color CCD camera, lighting unit, and ImageTool software, they extracted kernel dimensions (length, perimeter, projected area) from gray-scale images, calibrated with brass disks. The study found a power-law relationship between HRY and kernel dimensions, with the projected area yielding the most accurate HRY estimation (RMSE < 2%). Discrepancies were noted between HRY obtained from the rice grader and image analysis, likely due to grader imprecision. Results underscore the potential for precise image-based DOM and HRY monitoring in milling quality assessment.

3. Existing Stipulations

i. Indian Standard IS 4333 (Part IV) - determination of the mass of 1000 grains

This standard outlines procedures for assessing rice quality, specifically determining the degree of milling by measuring weight loss. Identical to ISO 520:1977, 'Cereals and pulses — Determination of the mass of 1000 grains,' this Indian Standard was adopted by the Bureau of Indian Standards. The principle involves weighing a sample, separating whole grains, and weighing the residue. The mass of whole grains is then divided by their number, with the result expressed in relation to 1000 grains, either on a wet or dry basis. A random amount, approximating the mass of 500 grains, is weighed to the nearest 0.01 g. The number of grains is calculated using a photoelectric counter, or manually if suitable equipment is unavailable.

Mass of 1000 grains is calculated by following formula

$$Mh = \frac{Mo\ x\ 1000}{N}$$

Where,

Mh = mass of 1000 grains

Mo = the mass, in grams, of the whole grains;

N = the number of whole grains in the mass Mo

For dry basis mass Ms of 1000 grains

$$Ms = \frac{Ms(100 - H)}{100}$$

Where,

H= moisture content of rice

The calculations are performed both before and after milling, and the percentage difference between them is used to determine the degree of milling."

ii. Indian Standard IS 4333 (Part IV) - determination of dehusked grains in rice by double staining method

In this method, 5 g of rice (including sound head rice and brokens) is placed in a petri dish and dipped in 20 ml of methylene blue solution, allowing it to stand for about 1 minute. The methylene blue solution is then decanted, and the grains are given a swirl wash with 20 ml of dilute hydrochloric acid, followed by a water rinse. Next, 20 ml of metanil yellow solution is poured over the blue-stained grains and left to stand for 1 minute. The effluent is decanted, and the grains are washed twice with fresh water. The residual bran or milled rice kernels will stain green, while the rest of the kernels remain yellow. The stained grains are kept under fresh water, and the de-husked grains (indicated by the green stain) are counted. The total number of grains in the 5 g sample is also counted, with three broken grains considered equivalent to one whole grain.

Percent of de-husked grains = $\frac{N\ x\ 100}{W}$

Where, N = number of de-husked grain in 5 g of sample,

and W = total grains in 5 g of sample.

iii. ISO 6646:2011 - Rice — Determination of the Potential Milling Yield from Paddy and from Husked Rice

In this method, a test sample is divided into a portion suitable for the equipment, passed through an aspiration apparatus to remove light matter, and spread to eliminate any extraneous material. The sample is then weighed to the nearest 0.01 g, ensuring a minimum of 200 g. The paddy is dehusked using a testing husker, with any undehusked grains manually separated and dehusked again. The residual paddy is also manually dehusked and added to the husked rice, which is weighed to the nearest 0.01 g. A portion of the husked rice, with a minimum weight of 100 g, is then divided and weighed. The testing mill is thoroughly cleaned, and the husked rice sample is milled for a time predetermined by trials to remove a specific mass fraction. The milled rice is weighed, and the head rice is separated from the broken kernels, with both fractions weighed and recorded to the nearest 0.01 g.

Calculation of milling yields

Parameter	Paddy	Husked rice
Husked rice, y_0	m_y/m_x	1
Milled rice, y_1	m_1/m_z	m_1/m_z
Milled head rice, y_2	m_2/m_z	m_2/m_z

Where,

m_x = Sample weight of paddy (≥ 200 g.)

m_y = weight of husked rice

m_z = aliquot of husked rice (≥ 100 g.)

m_1 = milled rice

m_2 = milled head rice

Potential yield of husked rice, y_h

$$y_h = y_0 \times 100\ \%$$

Potential yield of milled rice, y_m

$$y_m = y_0 y_1 \times 100\ \%$$

Potential yield of milled head rice, y_{mh}

$$y_{mh} = y_0 y_2 \times 100\ \%$$

4. Sampling Method

The raw material is being collected from various rice mills located in different agroclimatic zones of India to ensure diversity in the experimental samples. The samples being taken for testing include white rice, brown rice, and parboiled rice. At least three different varieties of raw and parboiled paddy and corresponding rice are being collected from the states of West Bengal, Assam, Maharashtra, Chhattisgarh, Punjab, and Kerala.

Sample Collection Procedure

i. Selection of Mills

- Identify and select at least five rice mills in each zone to ensure a comprehensive representation of local varieties.

  Collection Method

- Samples are being collected directly from the mills or received through courier service.
- Each sample is being packed in airtight zip-lock bags to prevent moisture absorption and contamination.

ii. Storage Conditions

- All samples are being stored at temperatures below 25℃ until milling trials are conducted.
- The moisture content of all samples is being maintained between 11-13% wet basis (wb) to ensure stability during storage.

iii.Documentation

- Each sample collected is being labeled with details including the collection date, mill name, variety, and location.
- A sampling log is being maintained to track all collected samples and their respective details.

Quality Assurance

Periodic checks are being conducted to ensure that the sampling procedure adheres to the set protocols and that the integrity of the samples is maintained throughout the collection and storage processes.

5. Methodology

i. Sample Preparation and Milling

- Varieties: Utilize 30+ rice varieties from different climatic zones, polished at intervals of 15s, 20s, …, up to 60s to create varying DOM levels.

- Sample Size: Prepare 100g samples for each milling interval and rice variety, ensuring triplicates for consistency and repeatability.
- Storage: After milling, all samples will be stored below 5°C to prevent chemical changes like lipid rancidity, ensuring sample integrity throughout the project.

ii. Physical Characterization

- Density and Porosity: Measure true and bulk density using a gas pycnometer and calculate porosity.
- Whiteness Index and Color Measurement: Use a whiteness meter and a Hunter Lab colorimeter to evaluate the optical properties of the rice.

iii. Chemical Analysis

- Fat Content: Determine fat content in polished rice using Soxhlet extraction, conducting each measurement in duplicates for validation.
- Starch Content: Quantify starch in bran using the iodine test, performing duplicate tests for each sample.
- Protein Content: Use the Kjeldahl method for protein analysis in white rice, with measurements duplicated for accuracy.
- Mineral Content: Assess mineral content in white rice, correlating these chemical properties with DOM levels.

iv. Image Sensing and Digital Analysis

- Imaging Setup: Employ a multi-camera system (RGB or NIR) to capture 360-degree images of each rice kernel.
- Image Processing: Utilize machine vision software to detect and measure bran areas, calculating DOM based on the ratio of bran area to total surface area.

6. Validation Plan Aligned with ISO 5725

i. Accuracy (Trueness) Validation

- Procedure: Compare the developed method's results with established reference methods (e.g., gravimetric analysis) across multiple rice samples and milling intervals. Adjust the method based on any bias observed.
- ISO 5725 Compliance: Ensures trueness by verifying against known standards.

ii. Precision Validation

- Repeatability: Conduct tests on each sample in duplicates under identical conditions to ensure consistency.
- Reproducibility: Perform tests across different labs, using the same procedures and rice varieties, involving different operators.
- ISO 5725 Compliance: Evaluates both repeatability and reproducibility for precision.

iii. Statistical Analysis

- ANOVA: Used to detect significant differences in DOM values across samples and labs.
- Correlation: Pearson's correlation to assess relationships between DOM, chemical properties, and physical attributes.
- ISO 5725 Compliance: Statistical analysis validates precision and consistency.

iv. Inter-Laboratory Validation

- Procedure: Test the method in multiple laboratories with identical protocols, ensuring reproducibility across settings.
- ISO 5725 Compliance: Confirms method robustness in diverse conditions.

7. Progress Summary for the First Month

i. ***Staffing Update:*** Two junior project assistants have been hired for the project. The newly recruited staff joined on 01.11.24.

ii. ***Sample Acquisition:*** Over 10 raw and parboiled varieties have been collected from the state of Assam, Kerala, and Odisha. In the second month, twenty more samples (raw and parboiled each) will be collected

iii. ***Sample Preparation:*** The collected paddy varieties were cleaned using a laboratory-scale aspirator and stored under controlled temperature condition.

iv. ***Chemical Procurement:*** All required chemicals for conducting chemical tests (e.g., fat, starch, protein) have been procured.

8. Road map

Stage	Activity	1st month	2nd month	3rd month	4th month	5th month	6th month
I	Literature review and market survey on milling study and paddy varieties respectively.	✓					
	Procurement of common and grade A paddy varieties grown and processed (raw and parboiled) across India.						
	Conduct milling trials to obtained brown rice and polished (white) rice.						
II	Conduct quality analysis of bran sample and corresponding rice sample to explore the feasibility of deploying any chemical method.						
	Submission of interim report to Sectional Committee						
	Conduct light scattering and absorbance-based trials on the milled samples using a simple and indigenously developed illumination sensor.						
	Statistical analysis and optimization of obtained results from the experimental trials.						
III	Procurement of 50 plus commercially available raw and parboiled rice samples to validate the obtained results						
IV	Report submission to Sectional Committee for the evaluation and possible recommendation for further changes.						

References

1. Chen, W., Li, W., and Wang, Y. (2022). "Evaluation of Rice Degree of Milling Based on Bayesian Optimization and Multi-Scale Residual Model." *Foods*, 11, 3720.

2. Kale, S. J., Jha, S. K., and Nath, P. (2019). "Determination of Optimum Degrees of Milling for Raw and Parboiled Basmati (PB1121) Rice Using Principal Component Analysis." *Journal of Agricultural Engineering*, 56 (4), 235-248.

3. Lamberts, L., Bie, E. D., Vandeputte, G. E., Veraverbeke, W. S., Derycke, V., Man, V. D., and Delcour, J.A. (2007). "Effect of Milling on Colour and Nutritional Properties of Rice." *Food Chemistry*, 100, 1496–1503.

4. Mardison, Sutrisno, Usman, A., and Slamet. (2018). "Mathematical Model to Determine Rice Milling Degree Based on Absorbance Characteristic of Rice Solution at UV Spectrum." *International Journal of Scientific & Technology Research*, 7(12), 76-80.

5. Miller, B.S., Lee, M.S., Pomeranz, Y., and Rousser, R. (1978). "A Rapid, Objective Method to Measure the Degree of Milling of Rice." *Cereal Chemistry*, 56(3), 172-180.

6. Paul, H., Nath, B.C., Bhuiyan, M.G.K., and Shozib, B. (2019). "Effect of Degree Of Milling On Rice Grain Quality." *Journal of Agricultural Engineering*, The Institution of Engineers, Bangladesh, Vol. 42/AE, Number 4, December 2019, pp. 69-76.

7. Wan, P., and Long, C. (2010). "An Inspection Method of Rice Milling Degree Based on Machine Vision and Gray-Gradient Co-occurrence Matrix." *4th Conference on Computer and Computing Technologies in Agriculture (CCTA)*, Nanchang, China, pp. 195-202.

8. Yadav, B.K., and Jindal, V.K. (2001). "Monitoring Milling Quality of Rice by Image Analysis." *Computers and Electronics in Agriculture*, 33, 19–33.