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Robust NIRS Models for Non-Destructive Prediction of Physicochemical Properties and ageing of Basmati Rice

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

Aim: To determine physicochemical properties and age of rice by non-destructive technique.
Place and Duration of Study: Study was conducted at Division of Food Science and Postharvest Technology, Indian Agricultural Research Institute, New Delhi during 2020 to 2021.
Methodology: Rice were kept for accelerated aging at 42.6°C temperature & 71% RH for a duration of 30 days. Changes in four physicochemical properties namely amylose content, volume expansion ratio (VER), water absorption ratio (WAR), and kernel elongation ratio (KER) were

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evaluated destructively (by spectrophotometer and cooking method) and non-destructively (by spectroradiometer) at every alternate day, during 30 days storage.
Results: The physicochemical parameters of rice showed a good correlation with spectral signatures. Subsequently, Principal component Analysis (PCA), Partial Least Square Regression (PLSR), and Multiple Linear Regression (MLR) were used to model the physicochemical changes occurring during the process of accelerated aging using spectral reflectance values. Based on values of Coefficient of determination (R^2) and Root mean square error (RMSE) accuracy of models was determined. Predictions with the MLR model resulted in a coefficient of determination (R^2) of 0.82, 0.87, 0.9,7, 0.83 and 0.82 with root mean square error (RMSE) of 0.18, 0.13, 0.21, 0.124 and 4.2 for amylose content, VER, WAR, KER, and ageing process respectively for calibration.
Conclusion: The study demonstrated the potential of NIRS in non-destructively predicting the physiochemical parameters of rice.

Keywords: Accelerated ageing; basmati rice; non-destructive; reflectance; robust; spectroradiometer.

ABBREVIATIONS

MLR	*:*	*Multiple Linear Regression*
PSLR	*:*	*Partial Least Square Regression*
WAR	*:*	*Water Absorption Ratio*
VAR	*:*	*Volume Expansion Ratio*
KER	*:*	*Kernel Elongation Ratio*
PB	*:*	*Pusa Basmati*
PCA	*:*	*Principal Component Analysis*

1. INTRODUCTION

Rice (Oryza sativa L.) is a global staple food, especially in South Asia, with India as a major producer after China. It is primarily grown during the kharif season (June to September) but produced year-round in select regions. Rice consumption is significant in Arabian and South Asian countries, comprising carbohydrates, starch, and various nutrients. Basmati, a fragrant rice variety, is crucial in India, leading in production and export, serving as a significant crop for the country's economy [1].

Rice undergoes beneficial aging after harvesting, resulting in changes in physicochemical properties. Aging criteria are established based on physical and chemical properties, but the exact mechanism remains partly understood. Natural aging involves storing rice at room temperature for 3-6 months, a time-consuming process. Accelerated aging, where rice is stored at elevated temperatures for minutes to days, offers a more efficient alternative. Temperature, humidity, and storage duration are key factors impacting rice quality during aging [2].

Aged rice is preferred by Indian and Arabian consumers for its improved texture and premium qualities, especially in the case of basmati rice known for its aroma, long grains, and excellent cooking properties. Accurate assessment of rice aging is crucial to maintain quality and prevent fraudulent practices. Traditional methods for assessing rice aging are complex, time-consuming, and labor-intensive, requiring expensive equipment. Modern food industries demand quick, portable, and non-destructive techniques for quality determination [3].

Visible near-infrared (VNIR) spectroscopy is a valuable non-destructive technique widely used across industries like agriculture, cosmetics, food, polymers, pharmaceuticals, and textiles due to its speed, cost-efficiency, and minimal labor requirements [4]. VNIR spectroscopy can assess commodity quality in the field and during processing, relying on the scattering and absorption of light. Scattering reflects external properties like particle size and density, while absorption reveals the chemical composition [5].

VNIR spectroscopy is most successfully used for variety discrimination, determination of moisture content, pH, acidity, Various sugars, various diseases/rots, and evaluation of internal quality [6]. VNIR spectroscopy works in the wavelength range between 350- 2500 nm. VNIR spectrometer takes signature in terms of reflectance, absorbance, and transmittance [7]. VNIR spectroscopy is used for non-destructive evaluation of rice i.e amylose content of milled rice [8,9] quality of rice starch [10] textural properties of rice [11] quality characteristics of rice [12] evaluation of rice wine in terms of soluble solid and pH [13].

This experiment utilized VNIR spectroscopy in reflectance mode to assess the aging of various basmati rice varieties. The spectroradiometer's advantages include easy monitoring of the aging process and improved rice grading based on age, potentially increasing market value. The study aims to non-destructively analyze rice age using a VNIR spectroradiometer, with objectives to characterize artificially aged rice grains through spectroscopy and physical parameters and to develop models for discriminating and estimating aging in different rice grains.

2. MATERIALS AND METHODS

2.1 Procurement of Paddy

Freshly harvested four varieties of basmati rice [Pusa Basmati (PB)- 1121, PB-1509, PB- 1637, PB- 1718] and one non-basmati variety [*Pusa Sugandh* (PS)] were procured from Seed Production Unit, ICAR- Indian Agricultural Research Institute, New Delhi. Which was grown in the *kharif* season (June to October).

2.2 Sample Preparation

All five varieties of paddy namely PB- 1121, PB-1509, PB- 1637, PB- 1718, and PS were hulled in Satake rice huller (Satake Japan). After hulling brown is rice fed to a Satake rice polisher for polishing up to 6%. Brokens were separated and only head rice was used for further work.

2.3 Accelerated Ageing of Rice

For accelerated ageing freshly harvested milled rice of varieties PB 1121, PB 1509, PB 1718, PB 1637, and Pusa Sugandh were kept in a controlled chamber maintained at Relative humidity (RH) of 71 %, Temperature of 42.6 ºC for a period of 30 days as suggested by Rayguru et. al. [14] at National Phytotron Facility, IARI New Delhi.

2.4 Visible Near-Infrared Spectroscopy Analysis

Spectral signatures of fresh and accelerated aged samples (kept in a petri dish up to 1 cm thickness) were acquired before the destructive analysis in the wavelength range between 350-2500 nm at 1 nm intervals using a handheld spectroradiometer [Analytical Spectral Devise (ASD) Fieldspec® Spectroradiometer (350 to 2500 nm), Boulder, USA] at Hyperspectral Remote Sensing Laboratory, IARI, New Delhi. Care was taken to calibrate the device with a standard white plate before acquiring the spectral signatures. The precaution was also taken to ensure no gaps between grains to avoid signal losses. For each variety and accelerated storage period, six spectra were acquired to ensure the repeatability of the spectral signatures. A device was calibrated after every six spectra.

2.5 Determination of Physicochemical Properties of Rice

2.5.1 Amylose content

Amylose content in fresh and accelerated aged rice was determined using the spectrophotometric method suggested by Juliano (1971). 100 mg rice grains were grounded using mortar and pestle and transferred into the 100 ml volumetric flask. To it, 1 ml of ethanol and 10 ml of NaOH were added. The mixture was heated in a boiling water bath for 10 min. Distilled water was subsequently added to make the volume up to 100 ml. Out of 100 ml only 2.5 ml of the solution was then transferred into the 100 ml flask and 20 ml distilled water was added to it along with 3-4 drops of phenolphthalein indicator till it turns pink. Then 0.1 N HCl was added drop by drop until the pink color disappears. At last, 1 ml of iodine was added to it and the volume was made up to 50 ml using distilled water. The absorbance of the solution was measured at 510 nm and the amylose content was determined using a standard curve.

2.5.2 Volume expansion ratio

Volume expansion ratio (VER) was measured by using the toluene displacement method [15]. A specific volume of toluene was added to 250 ml of measuring cylinder then put 10 grains of uncooked rice and the initial reading was noted. Similarly, a change in the volume of 10 grains after cooking was also recorded.

$$\text{VER} = \frac{\text{Vc}}{\text{Vuc}}$$

Where,

Vc= Volume cooked rice

Vuc= Volume of uncooked rice

2.5.3 Water absorption ratio

Take 10 g of milled head rice and put it into 30 g of distilled water. Then beaker was kept in a boiling water bath (97 ± 2 ºC) for cooking. After cooking take the final weight of the rice [16].

$$\text{WAR} = \frac{\text{Wc}-\text{Wuc}}{\text{Wuc}}$$

Where,

Wc= Weight of cooked rice

Wuc= Weight of uncooked rice

2.5.4 Kernel elongation ratio

It is the ratio of the average length of grain after cooking to them before cooking. 10 grains of head rice were randomly selected from the sample and measured the length using a micrometer. Then grains were kept for cooking and measured for the final length [17].

$$\text{KER} = \frac{\text{Xc}}{\text{Xuc}}$$

Where,

Xc= Length of cooked rice

Xuc= Length of uncooked rice

2.6 Chemometric Analysis and Selection of Wavelength

For performing chemometric analysis different mathematical calculations and statistical methods were used. In chemometric analysis operations such as correlation, regression, calculation of the first derivative, differentiation of wavelength, development of model, and evaluation of model were done as described in the research work of various researchers [18].

The data available after the chemometrics was too large. The next step is to reduce the huge data in a particular manner and process only into selected data. Visible near-infrared (VNIR) spectroscopy wavelength ranges from 350 to 2500 nm were used in this study. The Visible Near-infrared reflectance spectra of rice samples shown in **Fig. 1**. In this huge range of wavelengths for each parameter, it is essential to select particular ranges of wavelengths because there are close relationships that can occur between wavelength and parameters. For the selection of wavelengths, correlation of physicochemical parameters namely amylose content, volume expansion ratio, water absorption ratio, and kernel elongation ratio with raw spectra as well as with the first derivative of reflectance were performed. These correlations were done by correlating four parameters with spectral reflectance data ranging from (351 to 2500 nm). As the correlation value ranges between -1 to 1, wavelengths showing high values of correlation were used for further analysis.

2.7 Multivariate Analysis

Multivariate analysis of the data was conducted to develop robust models for predicting the physicochemical parameters at different levels of accelerated aging and to predict the level of accelerated aging of rice. Spectral reflectance values acquired at all identified promising wavelengths were subjected to different available multivariate analyses like Partial least square regression (PLSR), principal component analysis (PCA), and multiple linear regression (MLR). Of the data used for multivariate analysis, 70% were utilized for model calibration, and the rest 30% for model validation. Maximum R^2 and minimum RMSE were considered for model selection.

3. RESULTS AND DISCUSSION

3.1 Amylose Content

The initial amylose content of rice varieties varied, with Pusa Sugandh at 22.21%, and among basmati types, PB 1718 had the highest at 23.19%, followed by PB 1509 (23.12%), PB 1121 (22.79%), and PB 1637 (22.60%). During 30 days of accelerated storage at 42.6°C, all varieties showed an increase in amylose content, with PB 1509 having the least change. This increase was most significant during the first 14 days, equivalent to 7 months of ambient storage. The elevated temperature likely caused amylopectin to convert into amylose, as observed in previous studies [19].

Amylose content significantly influences rice cooking quality. Higher amylose levels in aged rice improve cooking quality and processing efficiency by reducing stickiness and increasing grain firmness [20]. This is due to lower leaching of solids during cooking [21] and its positive correlation with water absorption, volume expansion, fluffiness, and grain separation. In the presence of lipids, amylose acts as both a diluent and a swelling inhibitor [22].

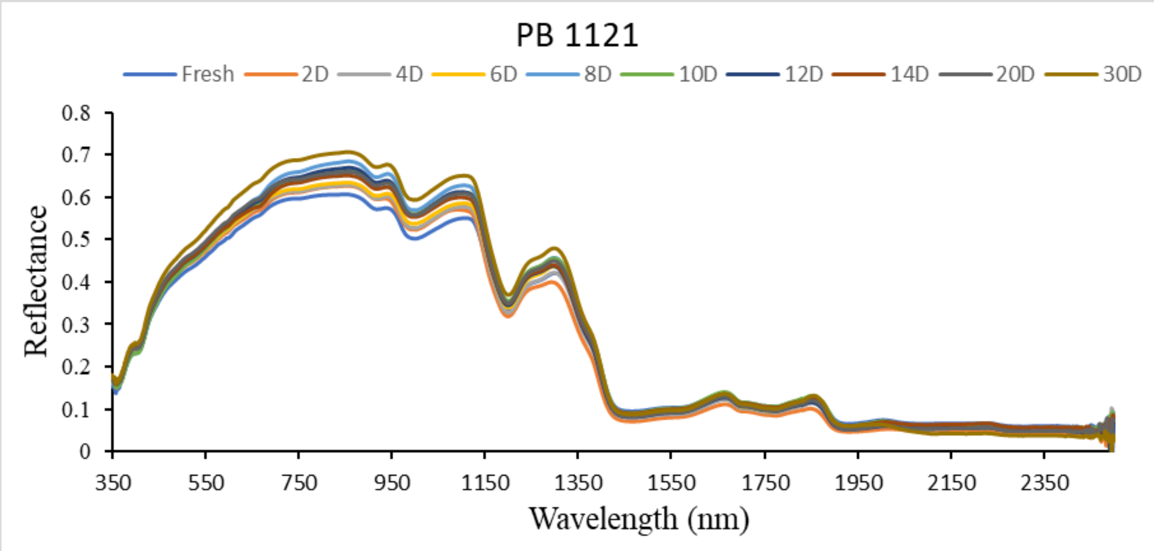
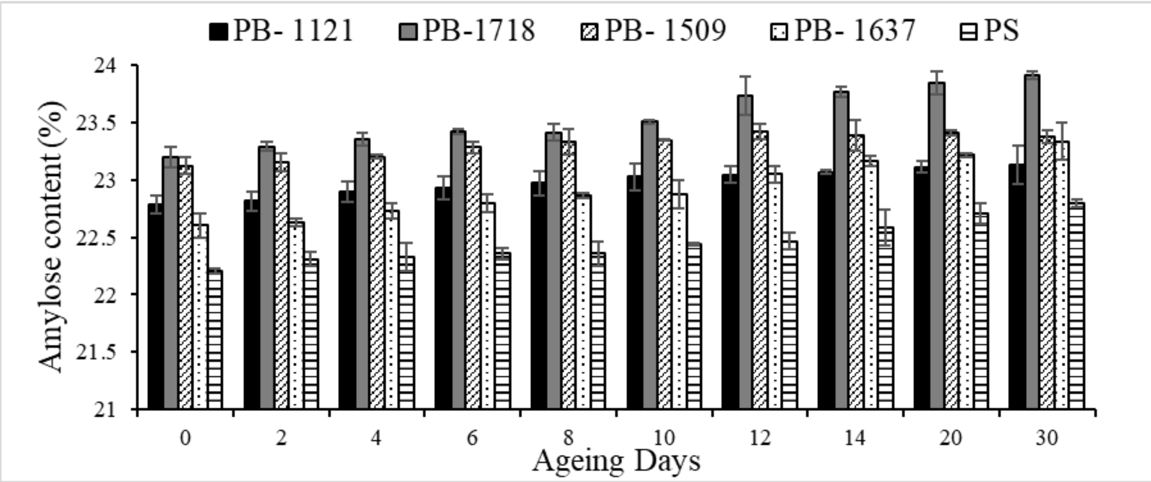


Fig. 1. Spectral signature of pusa basmati 1121 (PB 1121) after different periods of accelerated storage



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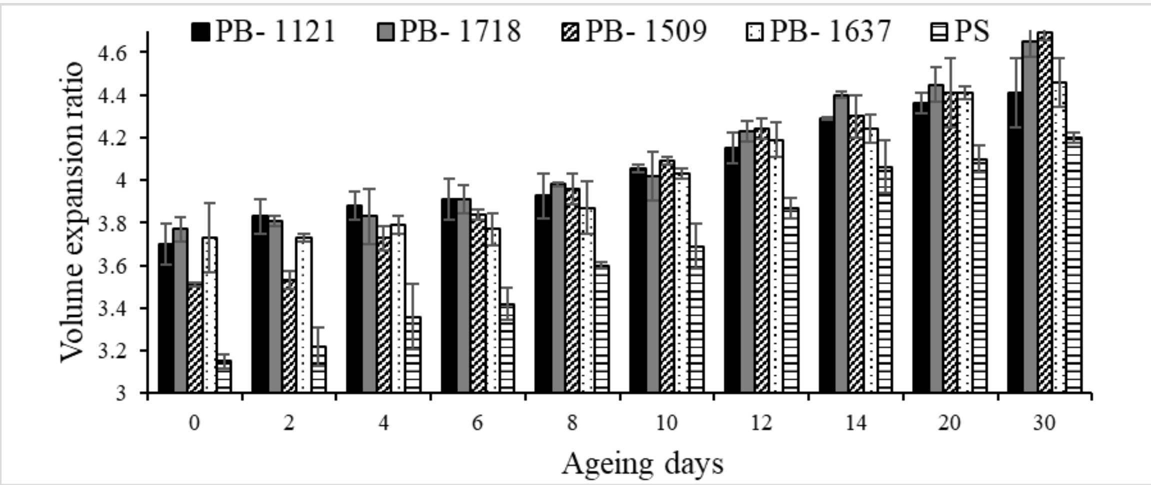

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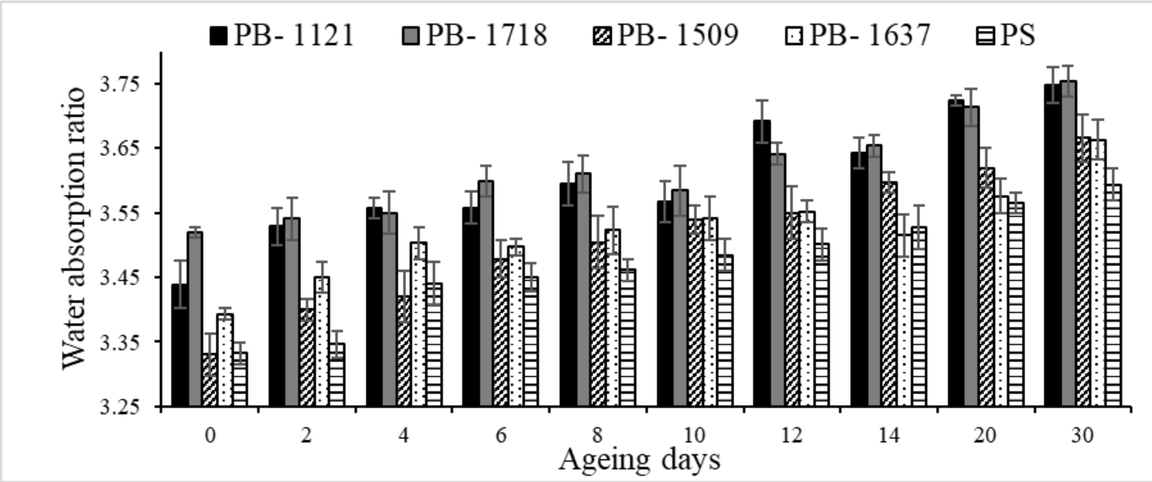

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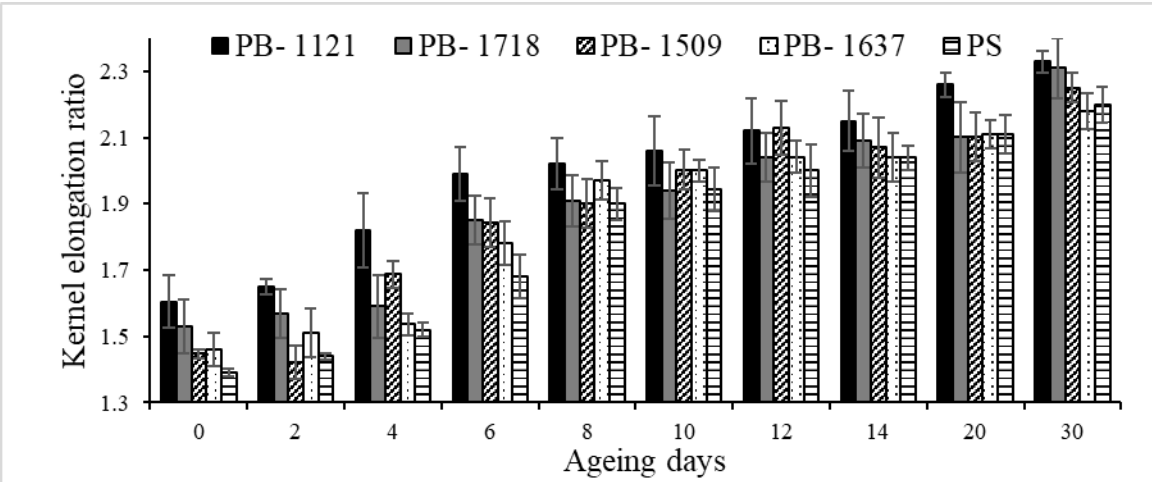


(d)

Fig. 2. Physiochemical changes in rice during ageing a) amylose content, b) VER, c) WAR, d) KER

3.2 Volume Expansion Ratio (VER)

Volume expansion ratio (VER) is crucial for rice cooking quality. During accelerated storage, all varieties showed increased VER. Pusa Sugandh had the lowest initial VER at 3.15, while basmati varieties ranged from 3.51 to 3.77. After 30 days, VER increased the most for PB 1509 (4.69), followed by PB 1718 (4.65), PB 1637 (4.46), PB 1121 (4.41), and Pusa Sugandh (4.20). This trend aligns with previous findings regarding rice aging and amylopectin content reduction, affecting pasting properties and increasing VER [20].

Accelerated storage induces structural changes in amylose chains, resulting in harder grains and higher Volume Expansion Ratio (VER). Consequently, cooked rice becomes firmer and less sticky due to increased water absorption and VER. Study by Indiarto & Nurannisa [23] also support the observed increase in VER during rice aging.

3.3 Water Absorption Ratio (WAR)

Water Absorption Ratio (WAR), an important cooking characteristic, mirrored the VER trend, as both are positively correlated due to increased water absorption contributing to higher volume expansion during cooking. Initial WAR values varied: PB 1121 (3.44), PB 1509 (3.33), PB 1637 (3.39), PB 1718 (3.52), and Pusa Sugandh (3.33). After 30 days of accelerated storage, WAR increased for all varieties, with PB 1121 at 3.75, PB 1509 at 3.67, PB 1637 at 3.66, PB 1718 at 3.75, and Pusa Sugandh at 3.59. Pusa Sugandh exhibited the least increase. Similar trends were reported by Zhou et al. [24] where aging led to increased water absorption due to rice hardness and amylose-lipid complex formation.

3.4 Kernel Elongation Ratio (KER)

Kernel Elongation Ratio (KER) is vital for understanding rice cooking and eating quality. PB 1121 had the highest KER at 1.62, significantly surpassing all other varieties, while Pusa Sugandh had the lowest at 1.39. Among basmati types, PB 1509, PB 1637, and PB 1718 had KER values of 1.45, 1.46, and 1.49, respectively. KER generally increased during 30 days of accelerated storage, reaching 2.32 (PB 1121), 2.25 (PB 1509), 2.17 (PB 1637), 2.31 (PB 1718), and 2.20 (Pusa Sugandh) on the 30th day. The aging effect on KER followed this order: Pusa Sugandh (58.27%), PB 1509 (55.17%), PB 1718 (55.03%), PB 1637 (48.63%), and PB 1121 (43.21%). These results align with findings from Indiarto & Nurannisa [23].

Variability in elongation ratio is linked to amylose content, particularly in PB 1121 and PB 1509, possibly due to amylose structure and its interaction with protein and lipids during accelerated storage. Starch granule expansion without disintegration depends on amylose content (Juliano, 1985), aligning with VER and WAR trends governed by structural changes and amylose content. Aging efficiency varies with rice variety, pre-treatments, techniques, and storage conditions [23].

3.5 Multivariate Analysis and Modeling

Spectral signatures were correlated with physicochemical parameters and aging duration, yielding correlation coefficients from -0.1 to 0.85 in the 400-1500 nm wavelength range. Correlation of raw spectral signatures with physicochemical parameters spanned wide wavelength ranges, making wavelength selection challenging. To address this, mathematical transformations, specifically 1st derivatives of spectral reflectance values, were employed. Correlation analysis revealed wavelength bands of interest: 1500-1800 nm for amylose content, 1200-1800 nm for VER, 1100-1800 nm for WAR, 500-1300 nm for KER, and 950-1400 nm for accelerated aging duration. For KER, correlation coefficients surpassed ± 0.8 at 1150-1350 nm. The derivatives demonstrated higher maximum correlation coefficients compared to raw spectral reflectance values (Table 1).

3.5.1 Amylose content

Amylose content changes during rice accelerated aging, affecting grain texture and cooking properties. PLSR models were developed using data in the 1500-1800 nm wavelength range, resulting in R^2 values of 0.65 (calibration) and 0.90 (validation), with an RMSE of 0.23. PCA showed that the first three principal components explained 94% of the variability. Subsequently, MLR modeling using sensitive wavelengths in the 1500-1800 nm range yielded R^2 values of 0.82 (calibration) and 0.84 (validation) with RMSE values of 0.186 and 0.210, respectively (Fig. 3a).

Matsau et al. [25] employed NIR spectroscopy, achieving an R^2 of 0.72 for amylose content in Japonica rice within the 850-1048 nm range. Fernández-Novales et al. [26] determined reducing sugars in grape ripening using Shortwave-NIR spectroscopy, obtaining an R^2 of 0.92 (800-1050 nm). Bao et al. [10] predicted rice starch quality with a spectroradiometer, yielding an R^2 of approximately 0.91 for amylose content (400-2500 nm). He et al. [27] used NIR spectroscopy for cereals, attaining R^2 values above 0.9 in the 1923-1961 nm range for starch and amylose content.

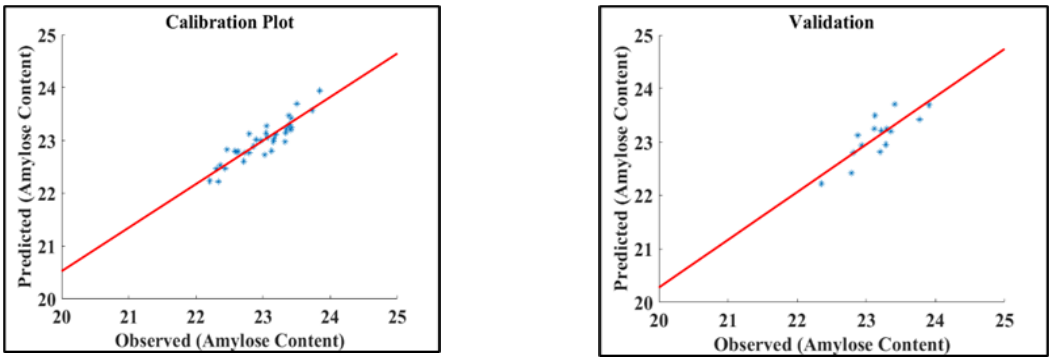
3.5.2 Volume Expansion Ratio

Similar to the amylose content approach, VER was correlated with spectral signatures. The first three principal components explained 94.6% of VER variability, with loading values peaking between 1200-1800 nm. PLSR models had R^2 values of 0.50 (calibration) and 0.67 (validation), with RMSE of 0.21. MLR models yielded R^2 values of 0.87 (calibration) and 0.85 (validation), with RMSE values of 0.13 and 0.15, respectively (Fig. 3b).

Moghimi et al., [28] attempted to develop calibration models for evaluating the TSS and acidity of kiwifruit. They developed a model using principal component analysis (PCA) and partial least square regression (PLS). The correlation coefficients for TSS and acidity were 0.93 and 0.943 respectively. RMSE values were 0.076% and 0.26°Brix respectively obtained between wavelength region of 400 to 1000 nm.

Table 1. Wavelengths showing maximum correlation with first derivative spectral reflectance

Parameter	Max. correlation	Wavelength range (nm)
Amylose content	0.7917	1500-1800
VER	0.7982	1200-1800
WAR	0.7630	1100-1800
KER	0.8463	500-1300



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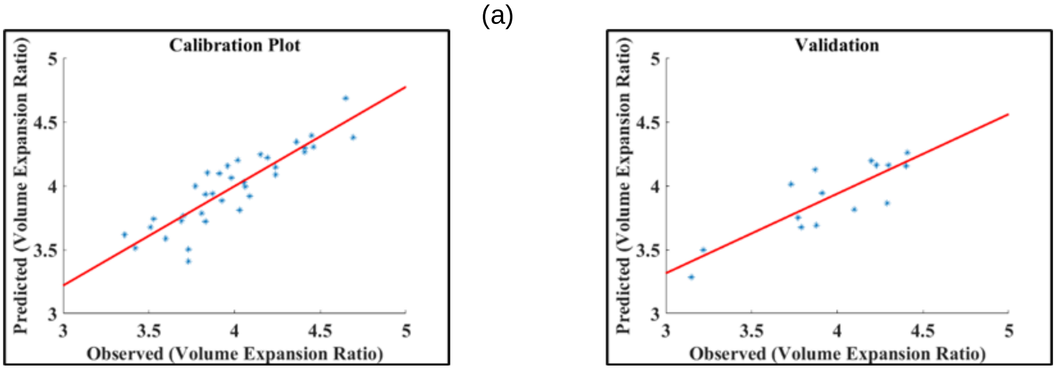

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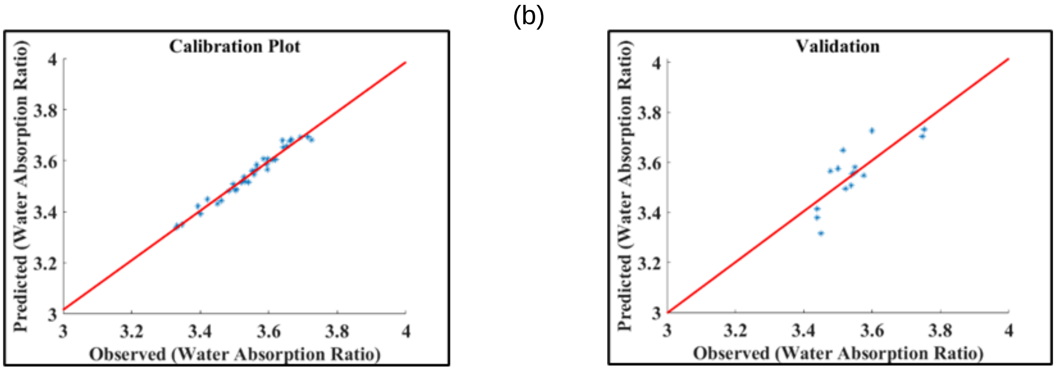

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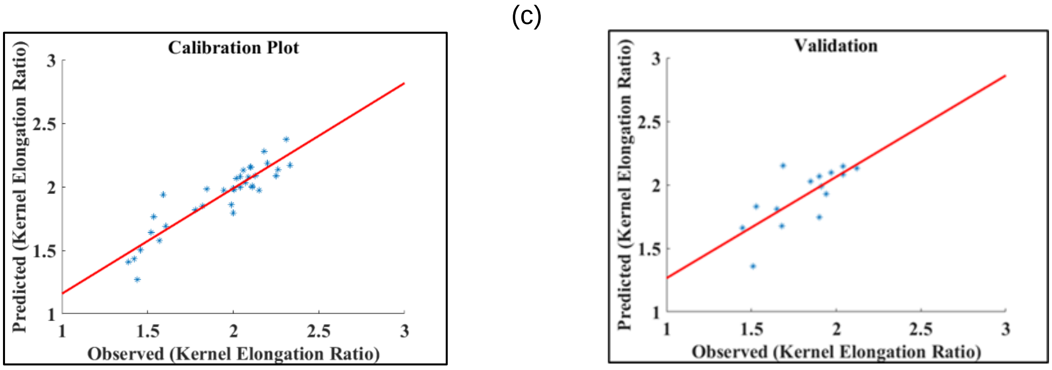

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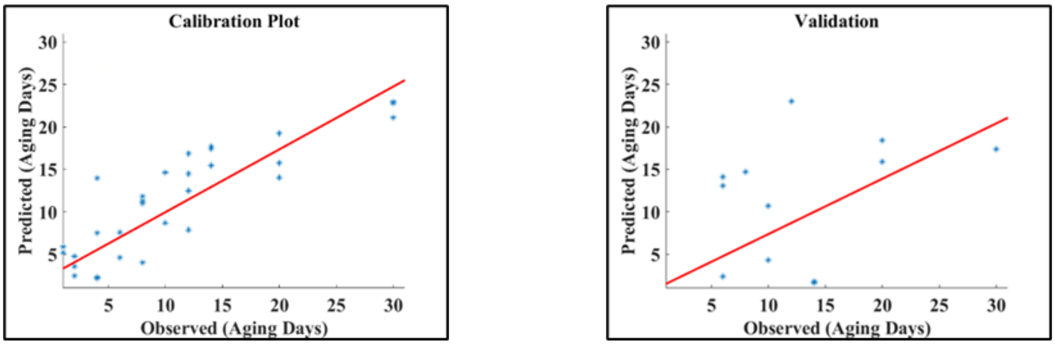

(e)

Fig. 3. MLR models for prediction of a) amylose content, b) VER, c) WAR, d) KER, e) ageing

3.5.3 Water expansion ratio

For water expansion ratio, the first three principal components explained 93.6% of the variability, with loading values peaking at 1100-1800 nm. PLSR models achieved R^2 values of 0.78 (calibration) and 0.70 (validation), with RMSE of 0.044. MLR models obtained R^2 values of 0.97 (calibration) and 0.90 (validation), with RMSE values of 0.21 and 0.25, respectively (Fig. 3c). In a study by Kusumiyati et al. [29] on apple fruit quality evaluation, NIR spectroradiometer yielded an R^2 of 0.81 and RMSE of 0.009 for water content within the 702-1065 nm wavelength band.

3.5.4 Kernel elongation ratio

For kernel elongation ratio (KER), the first three principal components explained 96% of the variability, with loading values peaking at 500-1300 nm wavelengths. PLSR models achieved R^2 values of 0.64 (calibration) and 0.84 (validation), with RMSE of 0.16 and 0.15. MLR models obtained R^2 values of 0.83 (calibration) and 0.85 (validation), with RMSE values of 0.124 and 0.11, respectively (Fig. 3d). In a study by Schmilovitch et al. [30] on mango physiological indices using NIR spectroscopy, MLR models had R^2 values of 0.92 for TSS and 0.6085 for acidity in the 1200-2400 nm wavelength region, outperforming PLSR models.

3.5.5 Ageing of rice

For accelerated aging, spectral signatures were correlated similarly to amylose content. The first three principal components explained 96% of the variability. PLSR models achieved R^2 values of 0.42 (calibration) and 0.58 (validation), with RMSE of 6.27. MLR models obtained R^2 values of 0.82 (calibration) and 0.70 (validation), with RMSE values of 4.2 and 4.5, respectively (Fig. 3e). In a study by Fernández-Novales et al. [26] on wine quality prediction during aging using NIR spectroscopy, it was found to be a promising technique for assessing grape wine quality attributes during fermentation and aging.

The chemometric analysis indicated that spectral reflectance values between 350 to 2500 nm could adequately predict quality. MLR outperformed PLSR in terms of R^2 and RMSE in both calibration and validation. MLR's superior performance is attributed to its selective wavelength choice, eliminating overfitting and collinearity issues observed in PLSR models (Table 2) (ElMasry et al., [31,32] Fernández-Novales et al., [26].

Table 2. Summary of PLSR and MLR statistics for amylose content, VER, WAR, KER, and ageing

Parameters	MLR Model				PLSR Model			
	Calibration		Validation		Calibration		Validation	
	R^2	RMSE	R^2	RMSE	R^2	RMSE	R^2	RMSE
Amylose content	0.82	0.18	0.84	0.21	0.64	0.23	0.90	0.23
VER	0.87	0.13	0.85	0.15	0.50	0.21	0.67	0.21
WAR	0.97	0.21	0.90	0.25	0.77	0.04	0.70	0.04
KER	0.83	0.124	0.85	0.11	0.63	0.15	0.84	0.15
Ageing	0.82	4.2	0.70	4.5	0.42	6.27	0.57	6.27

4. CONCLUSION

Physicochemical changes during accelerated rice aging were assessed both destructively and non-destructively. Spectral reflectance, including first derivatives, correlated with these changes and aging periods. PCA, PLSR, and MLR were used to develop predictive models.

- Spectral reflectance effectively captures amylose content, VER, WAR, and KER changes during rice aging, enabling age determination.
- Variabilities in physical parameters and aging were characterized within 600-1800 nm using raw spectral reflectance, and 900-1350 nm using 1st derivatives.
- MLR models outperformed PLSR models, achieving $R^2 > 0.80$ for physical parameters and age prediction (PSLR R^2: 0.42-0.77).

4.1 CRediT Authorship Contribution Statement

Patil Rajvardhan Kiran: Experimental analysis, Collecting dataset, Investigation, Original draft preparation. Abhijit Kar: Conceptualization, Methodology. Rabi Narayan Sahoo: Conceptualization, Methodology. Arunkumar T. V.: Investigation, Review and editing.

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COMPETING INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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