Chapter 3

3. Material and Methods

This chapter gives a detailed description of all the materials and methods used and implemented to fulfill our objectives. A pictorial view of the overall frame of the workflow of the complete methodology is given in Fig. 3.1. The first objective includes studying the kinetics of the parboiling process for *Chokuwa* rice of Assam, finding the conditions for the desired quality of parboiling. The second objective incorporates the design and development of an NIRS-based process analysis tool using ML models for determining the quality parameters of the parboiling process during the production of *Komal Chaul*. The third objective comprehends the study of ageing of parboiled *Chokuwa* rice (*Komal Chaul*) as well as non-parboiled *Chokuwa* rice and how storage and ageing affect its cooking quality. Fourthly, an NIR-based tool for predicting the degree of ageing and its cooking time (softening time).

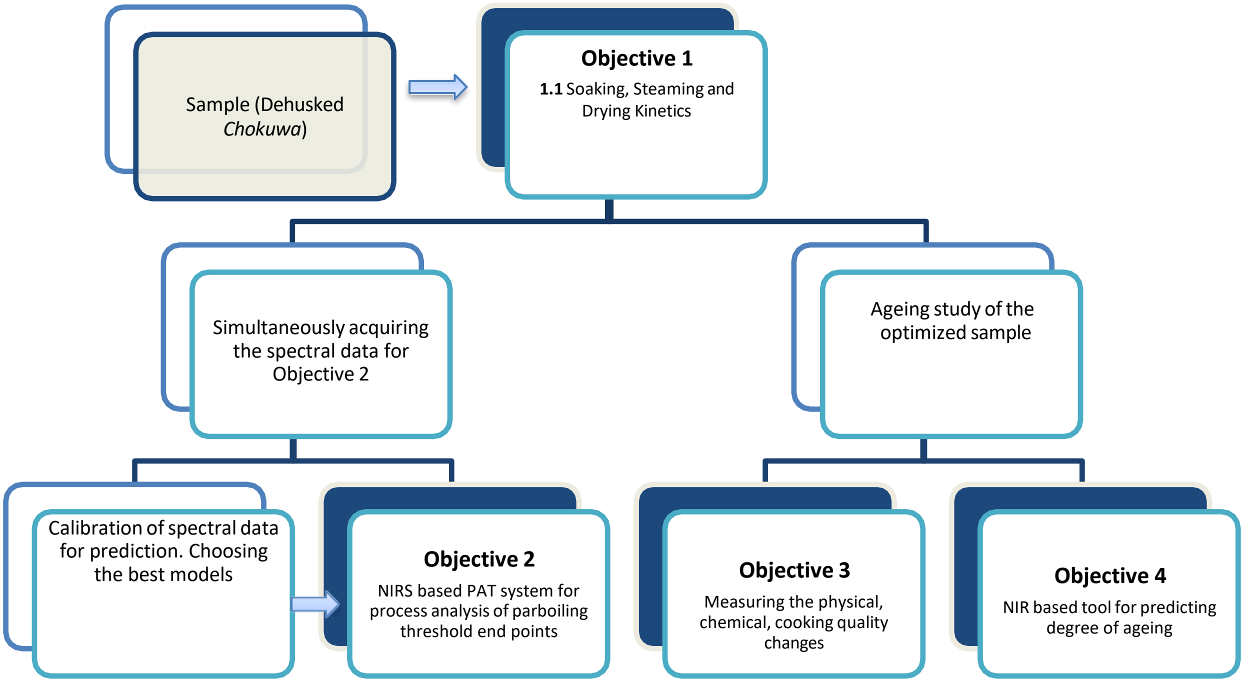


Fig. 3.1: Process flowchart describing the overall framework of the work

3.1 Materials

3.1.1 Raw Materials

Chokuwa paddy was first collected in the year 2019 from the Rice Research Station at Titabor, Assam. Afterward, for 3 consecutive years, freshly harvested paddy samples were collected from a farm in Jamugurihat, Assam (Fig. 3.2). The initial moisture content was around 16% (wb). So, initially, the paddy samples were dried to a moisture content of around 12-13% (wb) and stored in HDPE containers to avoid insect infestation. *Chokuwa* rice samples were dehulled and then kept in PVC bags at room temperature (Fig.3.3).

Fig. 3.2: *Chokuwa* paddy of Assam

Fig. 3.3: *Komal Chaul* produced from *Chokuwa* rice

3.1.2 Instruments

The list of instruments, machines and components used along with their model numbers are detailed in Table 3.1.

Table 3.1: List of major instruments used

S.N.	Detail of Equipment/ instruments	Purpose	Name of Laboratory/ Department/ Institute
1.	Near-Infrared Spectrophotometer Model: SCiO 1.2 Make: Consumer Physics, Israel	Scanning the sample acquiring spectrum in the range 740-1050 nm	FET department, Tezpur University
2.	Laboratory grain scanner Model: Canoscan 9000F, Make: Mark-II, Canon, Europe	Scanned and determined the physical dimensions of the grain kernels	FET department, Tezpur University
3.	Scanning electron microscope Model: JSM-6390LV Make: Jeol, Japan	Determined the morphology of the samples	Physics Department, Tezpur University
4.	UV-Vis Spectrophotometer (Double beam system, Wavelength:190-1000 nm) Model: CE 7400 or 7500 Make: CECIL Instrument Limited, UK	Used to measure the absorbance of samples during %DG determination	FET department, Tezpur University
5.	Rapid Visco-Analyzer Model: MCR 72 Make: Anton Paar	Used for pasting properties/ measurements.	FET department, Tezpur University
6.	X-ray Diffractometer Model: D8 Focus Make: Bruker Axs Germany	Used for XRD pattern and crystalline structure/physical properties study	SAIC, Tezpur University
7.	Fourier transform Infrared. Spectrophotometer Model: Frontier IR, Make: Perkin Elmer, USA	Used for measurement of structural fingerprints of molecules in the samples	Chemistry Department, Tezpur University
8.	Kjeldahl machine Model: Classic DX VA Make: Pelican Equipment	Measurement of the crude protein content of the sample	FET department, Tezpur University
9.	Texture Analyzer Model: TA-HD Plus Make: Stable Micro Systems, UK	Measurement of the textural parameters of the sample	FET department, Tezpur University

10.	Low Temperature chamber Model: Thermotech, Make: Sanco, India Temperature: 4-60℃	Used for maintaining at a particular temperature, used for sample storage.	FET department, Tezpur University
11.	Hot water bath Model: BW-20G Make: Lab Companion, USA	Use to heat the samples at a particular water temperature	FET department, Tezpur University
12.	Differential Scanning Calorimetry Model: Make: Perkin Elmer, USA	Used for measuring thermal properties of rice	FET department, Tezpur University
13.	Anemometer Model: Airflow Instruments Anemometer LCA301 Make: UK	Used for measurement of the air velocity	FET department, Tezpur University
14.	Gas pycnometer Model: PYC-100A make: Porous materials inc. USA	Used to determine the true volume and true density of powders and bulk solids using high precision	FET department, Tezpur University
15.	Laboratory sheller Model: RTE-07 Make: A-GRAIN, India	To remove the outer husk part from the paddy for obtaining brown rice	FET Department, Tezpur University
16.	Laboratory autoclave Model: JSGW-1001 Make: India	Used for hydrothermal treatment (steaming process) of the samples at pressure	FET Department, Tezpur University
17.	Laboratory tray dryer Model: Armfield – UOP8-A Make: UK	Used for drying process step at a particular temperature range and air velocity	FET Department, Tezpur University
18.	Triad Spectroscopy Sensor Model: AS7265x Make: Spark fun, India	Used for the development of a Vis-NIR sensor	FET Department, Tezpur University
19.	Arduino Mega 2560 Model: EC-1167 Make: India	Used for the development of a Vis-NIR sensor	FET Department, Tezpur University
20.	ESP32 Development Board with A 3.5-inch screen Model: WT32-SC01 Make: India	Used for the development of a Vis- NIR sensor	FET Department, Tezpur University
21.	Gel electrophoresis set up. Page Ruler Prestained Protein Ladder	Used for the development of a Vis- NIR sensor	MBBT department, Tezpur University

3.1.3 Software

The lists of soft computing tools used for the purpose of data handling, data extraction, curve fitting, statistical calculations, and machine learning modeling are as follows:

- Microsoft Excel 2007
- Scio Lab phone develop kit/ PC app
- Web plot digitizer 2010
- Jupyter Notebook in Anaconda (Python 3.8)
- Google Colab online (Python latest version updated)
- IBM SPSS 25
- Origin Pro 8.5
- Image J

3.2 Methodology

The preliminary physicochemical analysis of the sample included:

i. **Moisture content:** Moisture content (m.c.) was determined by the gravimetric method of weight determination where water loss was measured by taking the mass of oven-dried sample. Moisture content on wet basis (wb) was calculated by using Eq. 3.1 and moisture content on dry basis was calculated using Eq. 3.2. Dried mass was calculated by putting the sample at 105 °C for 6-7 hours.

$$m.c.(wb)\% = \frac{Initial\ mass - Dried\ solid\ mass}{Initial\ mass} \times 100 \tag{3.1}$$

$$m.c.(db)\% = \frac{Initial\ mass - Dried\ solid\ mass}{Dried\ solid\ mass} \times 100 \tag{3.2}$$

ii. **Amylose content:** Amylose content was measured using the method developed by Juliano (1970) [1], also cited in various other works. For the measurement, 100 mg of ground rice samples were taken and added to 1 ml of distilled ethanol. Then 10 ml of 1 N NaOH was added and was left for 6 hours. Afterward, the volume was made to 100 ml in a beaker by adding distilled water. Then taking 2.5 ml of the extract, added about 20 ml of distilled water and then two drops of phenolphthalein reagent. The alkalinity was adjusted by titrating with 0.1 N HCl until the pink color disappeared. 1 ml of iodine reagent was added, and the volume was made to 50 ml and the absorbance values were noted down at 590 nm. Similarly, the standards were prepared at 0.2, 0.4, 0.6, 0.8, and 1 ml of the standard amylose solution; and absorbance values were recorded at the same wavelength. For blank, 1 ml of iodine reagent was added to 50 ml of distilled water. The absorbance values of the standard concentration were plotted against wavelength. The amount of amylose was calculated for the sample using the standard curve.

iii. **Fat content:** Crude fat was estimated by the method suggested by AOAC (2009) [2]. Fat was estimated using the Soxhlet method where the sample in a thimble was subjected to extraction method in a petroleum ether solvent, and later followed by the gravimetric method of mass estimation to calculate the percentage of fat.

iv. **Protein content:** Crude protein was estimated using the Kjeldahl method which involves digestion-distillation-titration suggested by AOAC (2009) [2]. The protein nitrogen fraction taken into consideration is 5.75.

v. **Bulk density:** Bulk density (ρ_b) was estimated by using the relation given in Eq.3.3 for which measurements were made by pouring around 20 g (bulk) of samples to a 100 ml measuring cylinder of inner radius 0.035 m and height 0.24 m and settling the level by gentle tapping for recording the exact volume.

$$\rho_b = \frac{Mass\ of\ grains\ (kg)}{Volume\ occupied\ (m^3)} \qquad (3.3)$$

vi. **True density:** True density (ρ_t) of grains samples was measured by employing a gas pycnometer (PYC-100A). A measured mass of grains (5 g approx.) was placed in the chamber, and after inserting pressure. The difference in the pressure of the void chamber and pressure after injecting gas is measured and this difference is calculated in terms of volume. Density is calculated by dividing the mass by volume.

vii. **Grain dimensions:** Grain dimensions were obtained using a Grain scanner.

3.2.1 Methodology for kinetic study of parboiling process and spectral calibration

The objective procedure involved studying the soaking, steaming, and drying kinetics for the parboiling of *Komal Chaul*. It also involves calibration of spectral points to process parameters like moisture content during soaking, percent degree of gelatinization, and moisture content during drying.

3.2.1.1 Soaking kinetics

Chokuwa rice's hydration behavior was studied at 40, 50, and 60 °C for producing *Komal Chaul*. The experiments were done by immersing 80 g of brown rice in 240 ml of distilled water and then placed in different test tubes. Then, at each temperature in isothermal conditions using a water bath (Model: BW-20G, Make: Lab Companion) brown rice was soaked over a period of 180 min. At every 10 min interval, 2 g of samples from each tube were taken out and placed in a moisture box, and then removing excess surface moisture from the sample, the box was weighed and placed in a hot air oven at 105 °C. Moisture content was measured using the gravimetric method suggested by AOAC [3]. The framework for developing a soaking kinetic model is represented in Fig. 3.1.1.

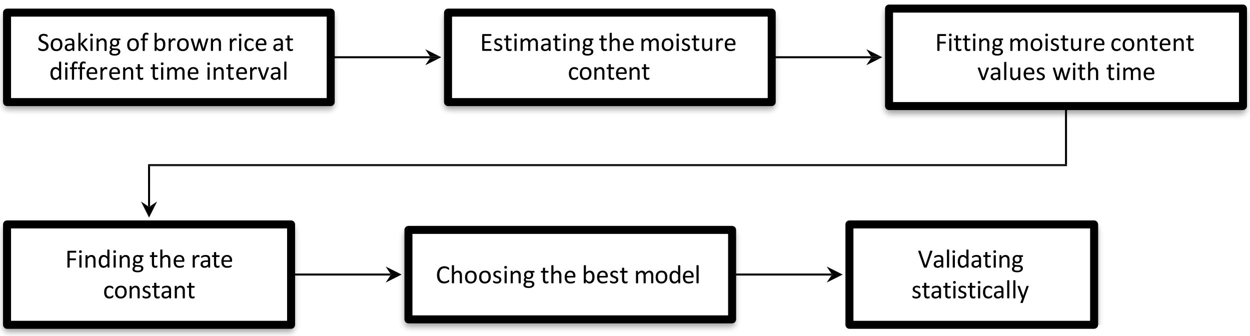


Fig. 3.1.1: Flow diagram for soaking kinetics

Peleg's equation was used to study the soaking kinetics (Eq. 3.4).

$$M(t) = M_0 \pm \frac{t}{(k_1 + k_2 t)} \tag{3.4}$$

where M is moisture content at any time M_0 is the initial moisture content (% db), t is time (min), k_1 (min/% db), and k_2 (1/% db).

The temperature dependence of $1/k_1$ followed an Arrhenius relationship and can be expressed as:

$$k = k_o e^{\left(-\frac{E_a}{RT}\right)} \tag{3.5}$$

where $k = 1/k_1$ (1/min) is the rate constant at any specific temperature, k_0 is the initial rate constant, E_a (kJ/kg mol) is the activation energy, T is the temperature, and R (kJ/kg mol K) is the universal gas constant. The rate constants (k) were estimated for each temperature and later the overall activation energy was calculated using the Arrhenius equation (Eq. 3.5).

And for hydration time,

As $$t \to \infty$$

$$M \to M_e(s)$$

And $$(M_e(s) - M_i) \to \frac{1}{k_2}$$

Thus, $$M_e(s) = M_i + \frac{1}{k_2} \quad (3.6)$$

Here, $M_e(s)$ is the equilibrium moisture content. $M_e(s)$ can be estimated by comparing equation 3.6. Saturation moisture content was also determined by the method suggested by Bello et al. [4].

$$M_s = \frac{M_i M_{i+2j} - M_{i+j}^2}{M_i + M_{i+2j} - 2M_{i+j}} \quad (3.7)$$

where M_i and M_j are the moisture content at times i and j.

This result was applied to each hydration range and averaged for each hydration temperature. These were used during the simulation to calculate the diffusion coefficients of water in brown rice.

Midili Kucuk equation (Eq. 3.8) was used to find the relation between time and moisture ratio and later the time shift factor (a_i) (Eq. 3.9) due to temperature was calculated simulating the time ratio (a_T) (Eq. 3.10) as a linear function of temperature. The developed temperature master curves were shifted to a reference temperature (50 °C) to construct a single master curve with the average temperature shift factor of a_T. This method was earlier suggested for the calculation of drying of turmeric slices and the developed generalized equation was later used for predicting moisture ratio at specific times and temperatures [5].

Generalization of Midili Kucuk equation:

$$MR = ae^{(-kt^n)} + bt \quad (3.8)$$

$$loga_i = log\ log\ t_R - logt_i \tag{3.9}$$

$$a_T = a_0 + a_1 T \tag{3.10}$$

The water absorption during the process of soaking brown rice at isothermal conditions was studied using Fick's diffusion equation (Eq. 3.11) assuming the coordinates to be spherical [6].

$$\frac{\partial C_A}{\partial t} = \frac{1}{r^2}\frac{\partial}{\partial r}\left(r^2 D_{C_A}\frac{\partial C_A}{\partial r}\right) \tag{3.11}$$

where, C_A is the volumetric concentration of diffusant A, D_{C_A} is effective diffusivity as a function of concentration, r is the radial coordinate, and t is time.

The moisture data obtained were calculated in terms of volumetric concentration using the following equation referred by Bello et al. [4]:

$$C_A = \frac{\rho_w \rho_s M}{\rho_w + \rho_s M} \tag{3.12}$$

Where, ρ_w and ρ_s and volume of pure water and pure solid (rice sample). M represents the local moisture content during soaking.

Equivalent diameter (d_p)was calculated using the formula:

$$d_p = l\frac{(w+t)^2}{4} \tag{3.13}$$

Where l is length, w is width, and t is thickness.

The initial assumptions that were taken into consideration to facilitate the calculations of this equation were:

1. The soaking process is isothermal.
2. Water diffusion into rice is one-dimensional, occurring in the radial direction.
3. The change in the volume of rice grain is equal to the volume of water absorbed. In order to solve this PDE (Eq. 3.11), the necessary initial condition (IC) and boundary conditions (BCs) were taken into consideration as proposed by Hsu et al. [7].

IC:

$$C_A = C_{A0}, \forall\, r, t = 0 \tag{3.14}$$

BCs:

$$\frac{\partial C_A}{\partial r} = 0, r = 0, t > 0 \tag{3.15}$$

$$C_A = \left[1 - e^{-\beta t}\right] C_{As} + C_{A0} e^{-\beta t}, r = 1, t > 0 \tag{3.16}$$

where, C_{As} is the saturation concentration of water in rice grain during the process of soaking and C_{A0} is the initial concentration of water before soaking.

The initial condition specifies that at t=0, the water concentration has an initial value at all points. The first boundary condition (Eq. 3.13) comes from the symmetry of spherical coordinates. The second boundary condition (Eq. 3.14) describes the change in concentration on the surface as a first-order equation with a rate constant β. β was estimated using experimental data.

Hsu et al. [7] also proposed the diffusion coefficient to be an exponential function of concentration (Eq. 3.17).

$$D_{\rho_A} = D_0 e^{\kappa C_A} \tag{3.17}$$

Where $D_0 and\ \ \kappa$ are arbitrary constants to be determined.

In order to avoid dimension issues during computation, the model was expressed in a dimensionless form of its variables. *S* is expressed as an integrative factor of C_A.

$$\Rightarrow \int_0^{C_A^*} D\, dC_A^* = \frac{(kC_A^* - 1)}{k} \tag{3.18}$$

$$R = \frac{r}{a} \tag{3.19}$$

$$T = \frac{D_0' t}{a^2} \tag{3.20}$$

In which:

$$C_A^* = \frac{C_A - C_{A0}}{C_{As} - C_{A0}} \tag{3.21}$$

$$k = \kappa (C_{As} - C_{A0}) \tag{3.22}$$

$$D = \frac{D_{AB}}{D_0'} \tag{3.23}$$

$$D_0' = D_0 e^{\kappa C_{A0}} \tag{3.24}$$

where, *k* and *κ* are arbitrary constants to be estimated.

Therefore, expressing volumetric concentration in Eq. (3.21) in a simplified form as,

$$C_A^* = 1 - e^{-BT}, \text{ at R=1} \tag{3.21*}$$

After changing it into a dimensionless form, the model, IC, and BC take the form:

$$\frac{\partial S}{\partial T} = D\left(\frac{\partial^2 S}{\partial R^2} + \frac{2}{R}\frac{\partial S}{\partial R}\right) \tag{3.25}$$

$$S = 0\ \forall R, T = 0$$

$$\frac{\partial S}{\partial R} = 0, R = 0, T > 0 \tag{3.26}$$

$$S = \int_0^{C^*} D dC^* = \frac{1}{k}(D - 1) = \frac{1}{k}\left[e^{kC^*} - 1\right] \tag{3.27}$$

Therefore, in a dimensionless and simplified form, (3.27*)

$$S = \frac{1}{k}\left[e^{\left[\left(k\left(1-e^{-BT}\right)\right)\right]} - 1\right], at\ R = 1, T > 0$$

Where $B = \frac{\beta a^2}{D_0'}$

Later, for computation the non-dimensional equation (Eq. 3.23) was discretized using the numerical method of finite difference. Here, it was done by approximating spatial derivatives by finite difference.

$$\frac{\partial S}{\partial R} \approx \frac{S_{i+1} - S_i}{\delta R} \tag{3.28}$$

$$\frac{\partial^2 S}{\partial R^2} \approx \frac{S_{i+1} - 2S_i + S_{i-1}}{(\delta R)^2} \tag{3.29}$$

Applying the approximations, the PDE became the following system of ordinary differential equations (ODEs):

$$\frac{dS_i}{dT} = (kS_i' + 1)\left[\frac{S_{i+1} - 2S_i + S_{i-1}}{(\delta R)^2} + \frac{2}{(i-1)\delta R}\left(\frac{S_{i+1} - S_i}{\delta R}\right)\right] \tag{3.30}$$

The initial and boundary conditions after discretization as ODE were:

$$\frac{dS_1}{dT} = \frac{dS_2}{dx}, R = 0, T > 0 \tag{3.31}$$

$$\frac{dS_{n+1}}{dT} = B\left[e^{\left[\left(k\left(1-e^{-BT}\right)\right)\right]} - BT\right], R = 1, T > 0 \tag{3.32}$$

The model calculations were done in Python. ODEs were solved using the Odient module from the Scipy library. The objective function (Eq. 3.32) was fitted to experimental values to obtain the constants D_0, κ, and β. The difference between the measured and the predicted were minimized by using particle swarm optimization.

Moisture ratios i.e., the amount of moisture measured at a time(t) to the amount of maximum absorbable moisture (M_∞) calculated using:

$$\frac{M_t}{M_\infty} = \frac{\int_0^1 C^* 4\pi R^2 dR}{\int_0^1 4\pi R^2 dR} \tag{3.33}$$

3.2.1.2 Steaming kinetics

The soaked brown rice was pressure parboiled placing it in a perforated vessel in an ICPD steam chamber (Appendix VII) of 3 l capacity at pressure varying from 0 - 0.2 MPa for different times ranging from 3-18 min. The steamed rice was dried at 55 °C for 8 hours and then made into powder of 200 mesh size. DG (%) was calculated by the method suggested by Birch et al. [8]. The procedure goes as 100 ml of distilled water was mixed with 0.2 g sample and treated with 2 ml of 10M KOH by stirring for 5 min and centrifuging for 25 min at 1500 rpm. After centrifugation, the sediment was discarded and from the supernatant 2 ml was pipetted out to dilute with 10 ml of distilled water, and then 0.1 ml of iodine solution was added. This procedure was repeated while keeping the amount of sample-KOH at 5 ml instead of 2 ml, and both their absorbance was measured at 600 nm in a spectrophotometer. The percent DG was then calculated as,

$$DG(\%) = \frac{Absorbance\ of\ sample\ (2\ ml)}{Absorbance\ of\ sample\ (5\ ml)} \times 100 \tag{3.34}$$

During steaming the condition $T_{Steam} > T_{gelatinization}$ was maintained, and the first-order gelatinization reaction was used as the model Eq (3.35).

$$\frac{dS}{dt} = -kS + C \tag{3.35}$$

Here, the un-gelatinized starch content ‘S’, determined from the DG study by amylose iodine method [8], at each time interval of steaming. Reaction rate constant *k*, and constant *C* was determined from regression analysis.

Therefore, it can be written as

$$\frac{dD_G}{(1-D_G)} = kdt \tag{3.36}$$

The earlier expression can be written as Eq. 3.37 by integrating Eq. 3.38 starting at 0 to t for time and from D_{G0} to D_G for DG values.

$$ln\left(\frac{1-D_G}{1-D_{G0}}\right) = kt \tag{3.37}$$

Therefore, taking exponential over the Eq. 3.35

$$D_G = 1-(1-D_{G0})e^{(kt)} \tag{3.38}$$

The steaming process can be defined using the mass transfer process and reaction rate using Eq. 3.39 [9]. Since, at a particular pressure the saturated steam temperature is constant, heat transfer change is assumed to be negligible.

$$\frac{dm}{dt} = D_{eff}\left(\frac{\partial^2 m}{\partial t^2} + \frac{2\partial u}{r\partial r}\right) - km \tag{3.39}$$

Where, D_{eff} = effective diffusivity, *m* = rice grain’s mean moisture content, *k* = reaction rate constant, *r* = equivalent radius of rough rice, *t* = steaming time at specific pressure.

The initial and boundary conditions are:

$$t = 0 \; and \; r = 0, m = m_0$$
$$t > 0 \; and \; r = 0, \frac{\partial m}{\partial r} = 0$$
$$t < 0 \; and \; r = r, m = m_e$$

where initial and equilibrium moisture contents are m_0 and m_e. Equilibrium moisture content can be calculated using the suggested by [10] as Eq. 3.40.

$$m_e = \frac{\left[\frac{c(T_{st}-T_{rt})}{\lambda} + 0.3\left(1+\left(\frac{C_{p0}T_{st}}{\lambda}\right)\right)\right]}{0.7\left[1+\frac{C_{p0}T_{rt}}{\lambda}\right]} \tag{3.40}$$

where C_{p0} is the specific heat of untreated brown rice can be calculated using Eq. 3.41 [11]. Specific heat C_p is calculated as a function of moisture content (M) for each part and then summed up as shown in Eq. 3.41, 3.42, and 3.43.

$$C_{p(Endorsperm)} = 1180 + 3766M \qquad (3.41)$$

$$C_{p(Bran)} = \frac{0.125}{\frac{1}{(1207 + 3807M)} - \frac{0.875}{C_{p(Endorsperm)}}} \qquad (3.42)$$

$$C_{p(Hull)} = \frac{0.2}{\frac{1}{(1109 + 4477M)} - \frac{0.1}{C_{p(Bran)}}} \qquad (3.43)$$

where, c-Hull=0.532, c-Bran=1.493, c-Endosperm=1.257.

Therefore, the solution for Eq. (3.39) with initial boundary conditions can be represented as follows:

$$Q = 8\pi r^3 D_{eff} m_e \left\{ \sum_{n=1}^{\infty} \frac{[kr^2 + D_{eff} n^2 \pi^2]kt}{[kr^2 + D_{eff} n^2 \pi^2]^2} - \sum_{n=1}^{\infty} \frac{[D_{eff} n^2 \pi^2]}{[kr^2 + D_{eff} n^2 \pi^2]^2} exp\left[-\left(\frac{[kr^2 + D_{eff} n^2 \pi^2]}{r^2} \right) t \right] + \sum_{n=1}^{\infty} \frac{D_{eff} n^2 \pi^2}{[kr^2 + D_{eff} n^2 \pi^2]^2} \right\} \qquad (3.44)$$

where the overall water absorbed at any t steaming time is Q.

3.2.1.3 Drying kinetics

The drying kinetics were studied at 3 different temperatures (40, 50, and 60 °C) for 180 min at time intervals of 10 min. The drying data were analyzed to provide a relation between moisture ratio and time. The semi-empirical models used are mentioned in Table 3.2.1.

Table 3.2: Semi-empirical models for studying drying

Model names	Equations	References
Page	$MR = e^{-kt^n}$	Laohavanich et al. 2008[12], Ertekin et al. 2005[13] and Mohapatra et al. 2005[14]
Newton	$MR = e^{-kt}$	
Modified Page	$MR = e^{-(kt)^n}$	
Henderson and Pabis	$MR = ae^{-kt}$	
Logarithmic	$MR = ae^{-kt} + c$	
Wang and Singh	$MR = 1 + at + bt^2$	
Thompson	$MR = alog(t) + b(log\ log\ (t)\ )^2$	
Diffusion	$MR = ae^{(-kt)^n}$	

The moisture ratio at any time ‘t’ was calculated using Eq. 3.45.

$$MR = \frac{\mathrm{M_t} - \mathrm{M_e}}{\mathrm{M_i} - \mathrm{M_e}} \qquad (3.45)$$

For the mechanistic approach, Fick's equation for a better understanding of moisture migration was used. For particles dried in layers of single grain, the drying kinetics was analyzed with formulation for particle-level moisture transfer. The analytical solution for spherical particles of Fick's second law of diffusion was represented as given in Eq. 3.46 [15].

$$MR = \frac{M - M_e}{M_o - M_e} = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} e^{\left(\frac{-D_{eff} n^2 \pi^2 t}{r^2}\right)} \tag{3.46}$$

Here, t is drying time, D_{eff} is effective moisture diffusivity, and r is the equivalent radius of the kernel (m). Equation (3.46.) will be simplified to only the first term of the series (Eq. 3.47 and Eq. 3.48) without affecting the accuracy of the prediction.

$$ln(MR) = \left(\frac{6}{\pi^2}\right) e^{\left(-\frac{D_{eff} \pi^2 t}{r^2}\right)} \tag{3.47}$$

or

$$\Rightarrow ln\, ln\left(\frac{M - M_e}{M_o - M_e}\right) = ln\, ln\left(\frac{6}{\pi^2}\right) - \left(\frac{D_{eff} \pi^2 t}{r^2}\right) \tag{3.48}$$

Diffusivity was calculated by fitting the linear plot of ln (*MR*) against *t.*

Further using the Hsu model for diffusivity calculations of hydration i.e., Eq. 3.18 to Eq. 3.24, a similar model was applied for drying diffusivity calculations. Due to moisture decrement during drying, in Eq. 3.21 and Eq. 3.23, C_{A0} and C_{As}were interchanged as follows:

$$C_A^* = \frac{C_{A0} - C_A}{C_{A0} - C_{As}} \qquad 3.21^*$$

$$k = \kappa(C_{A0} - C_{As}) \qquad 3.23^*$$

Midili Kucuk equation (Eq. 3.49) was used to find the relation between time and moisture ratio and later the time shift factor (a_i) (Eq. 3.50) due to temperature was calculated simulating the time ratio (a_T) (Eq. 3.51) as a linear function of temperature. The developed temperature master curves were calculated for a reference temperature (50 °C) to construct a single master curve with the average temperature shift factor of a_T. This method was earlier suggested for calculation of drying of turmeric slices and the developed generalized equation was later used for predicting moisture ratio at specific times and temperatures.

Generalization of Midili Kucuk equation:

$$MR = ae^{(-kt^n)} + bt \tag{3.49}$$

$$loga_i = log\, log\, t_R - logt_i \tag{3.50}$$

$$a_T = a_0 + a_1 T \tag{3.51}$$

3.2.1.4 Rehydration kinetics

The ready-to-eat *Komal Chaul* rehydration ability was studied using the same Hsu model (Eq. 3.18 – Eq. 3.24) used for hydration kinetics of soaking at 40, 50, and 60 °C. The saturation moisture was calculated using Peleg's equation calculation (Eq. 3.7). The activation energy was calculated using Peleg's constant ($1/k_1$) as represented in Eq. 3.5.

3.2.1.5 Calibration of processing parameters using spectra

Spectral values were obtained at each experimental point e.g., soaking data points at 40, 50, 60°C. The raw reflectance values were taken at each point using sensors. For, the process of soaking and steaming the NIR-based sensing device (SCiO 1.2, Consumer Physics) was used in the range of 740- 1100 nm. For drying, the laboratory-developed sensor Arduino Mega board-powered based AS7265x sensor was used. The raw reflectance values were converted to absorbance and integrating the area of the bands contributing largely for water gave us 'integrated absorbance' as suggested by Pervastch et al. [16]. This integrated absorbance was related to time that has a respective concentration. The average integrated absorbance values were then linearly expressed as a function of concentration. The same steps were followed for steaming and drying. Steaming the respective pressure points (0-0.2 MPa) at every time interval was considered and for drying likewise as described thoroughly in sections 3.2.1.1, 3.2.1.2, and 3.2.1.3.

3.2.2 Methodology for spectral calibration of parameters during parboiling using ML models

This objective's methodology was a continuation of objective 1. The methodology is divided into two parts: The first part estimated moisture level during hydration, degree of gelatinization during steaming, and moisture level during air drying for *Komal Chaul* production process using regression techniques like Principal Component Regression (PCR), Partial Least Square (PLS), and Artificial Neural Network (ANN). The second part was classifying the spectral values using threshold points of moisture measurement into classes for soaking and drying. Since steaming is done in a closed system endpoint classification wasn't feasible.

The overall methodology for implementation of spectral-based measurement is depicted in Fig. 3.2.1.

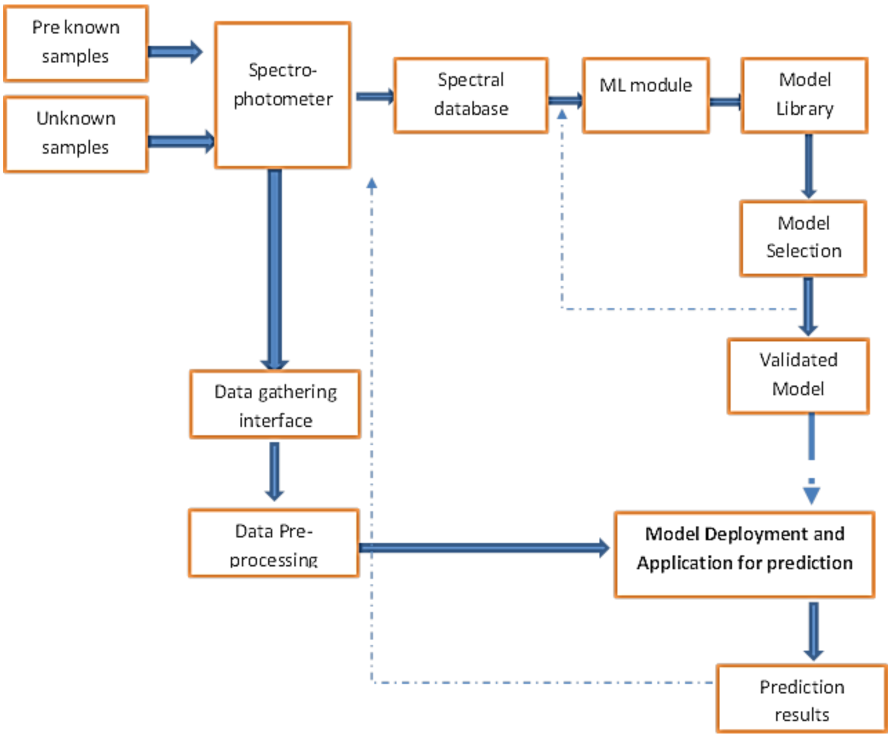


Fig. 3.2.1: Overall framework of PAT based spectral system

- **Spectrophotometer:** Spectral data were acquired using the portable NIR sensing device (SCiO 1.2) (Fig. 3.2.2). The NIR sensing device comes with a sample holder, liquid scanner, optical shade, and USB cable for charging. The solid sample was placed in the sample holder (Fig. 3.2.3) and above it the sensor was placed (Fig. 3.2.4). The phone application named 'SCiO Lab' was downloaded from Google Play store and the basic minimum login details were added. In SDK developer application in the phone Data Collection icon was tapped to add the new sample and then selected 'New Sample tab'. After entering the attributes of the new sample, they were scanned a minimum of three times. Lastly, 'Done' tab was selected when finished with all scans for that sample.

Fig. 3.2.2: Portable NIR sensing device (SCiO 1.2, Consumer Physics)

Fig. 3.2.3: Top view of sample holder

Fig. 3.2.4: NIR sensing device with sample holder

- **Data Preparation:** Spectral data (inputs) were arranged in .csv files in table format containing target variables against each spectrum. Spectral data were represented in the n×m matrix, where reflectance values for n number of samples for m wavelengths.

Before model application data needed to be handled or pre-processed. Data handling involved checking for errors and missing values. For continuous spectral data there was no chance for missing values and quality attributes for this work were numerical data. For categorical data, an ordinal scale was used. Then, data was properly wrangled with labels in a proper Dataframe. Ordinal and categorical data were standardized and then all the features were visualized for finding the distribution pattern using biplots or pair plots.

Lastly, for preprocessing logarithmic conversion followed by first-order derivative, SNV improves the PLS model prediction, especially when used on NIR data with scattering effects [17]. First-order and second-order derivatives are also used most of the time and it performs well. Ensemble preprocessing of NIR spectra for multivariate calibration performs better [18].

- **Choosing a Model:** Table 3.1 gives a detailed view of which model to choose based on our objectives.
- **Training the Model:** The dataset was divided into training set, validation set, and test set. K-fold cross-validation was used for splitting the data in k number of folds to reduce the variances among prediction accuracies.
- **Validating the Model:** The validation dataset was then used for validating the models using statistical measurement (Table 3.2). To avoid overfitting or underfitting, the model needs to be tuned, known as hyperparameter tuning. It includes the number of training steps, learning rate, initialization values, and error distribution using the test set. Therefore, the optimal hyperparameters were calculated based on minimum error.
- **Testing the model or Predictions:** Lastly, further test data that have been withheld from the model (and for which class labels are known), were used to test the model for a better approximation of how the model will perform in the real world. If the prediction error is too high the data was retrained or tested over a different model and the same procedure was repeated for training, validation, and testing.

3.2.2.1 Soaking endpoint prediction using spectral data

The framework of developing a soaking endpoint prediction model is explained in Fig 3.2.5. For this spectra-based model, the sampling chosen was similar to the one mentioned in section 3.2.1.1. to accurately predict the water absorption rate at different temperatures. At every temperature i.e., 40, 50, and 60 °C, and at every time interval of 10 min throughout the 0 – 180 minute soaking process, each spectrum was obtained in triplicates giving us a total scan of 171 (3 x 19 x 3). The spectra were collected using the NIR sensing device (SCiO 1.2). So, firstly the respective quality attributes were set, and labeling was done based on every category in SCiO Lab software. In SCiO Lab first, the data Collection icon was tapped to add the new sample and then select the New Sample tab. After entering the moisture content values that were measured values in the previous experiment in section 3.2.1.1, samples were scanned a minimum of three times. Lastly, the 'Done' tab was selected when finished with all our scans for that sample. The soaked rice scans were taken at 40, 50, and 60 °C and at every time 10 min. The spectra were obtained in the range of 740-1050 nm.

PLS was used as a regression model for estimating the quantitative value of moisture during the soaking of rice. The ML model was trained using the training data set. The k-fold function was used since the dataset consists of more than 100 spectra. To avoid biases, we split the data using k-fold cross-validation. Principal component regression was also trained, validated, and tested over the training dataset, validation dataset, and test dataset.

For classification, the spectral data was grouped into three classes based on moisture content *viz*. A (14-24%), B (25-30%) and C (>30%). Classification models like Logistic Regression, Linear Discriminant Analysis, K-Nearest Neighbors, Classification and Regression Tree, Naïve Bayes, Support Vector Machines, and Random Forest Classifiers. Weighted cross entropy (CE) (Eq. 3.52) and focal loss (FL) values (Eq. 3.53) were calculated to address the class imbalance problem for the minority class.

$$CE(p_t) = -\alpha_t log\ (p_t) \tag{3.52}$$

$$FL(p_t) = -\alpha_t (1 - p_t)^{\gamma} log\ (p_t) \tag{3.53}$$

Where, p_t is the probability of occurrence of that class, α_t =1 and $\gamma = 2$.

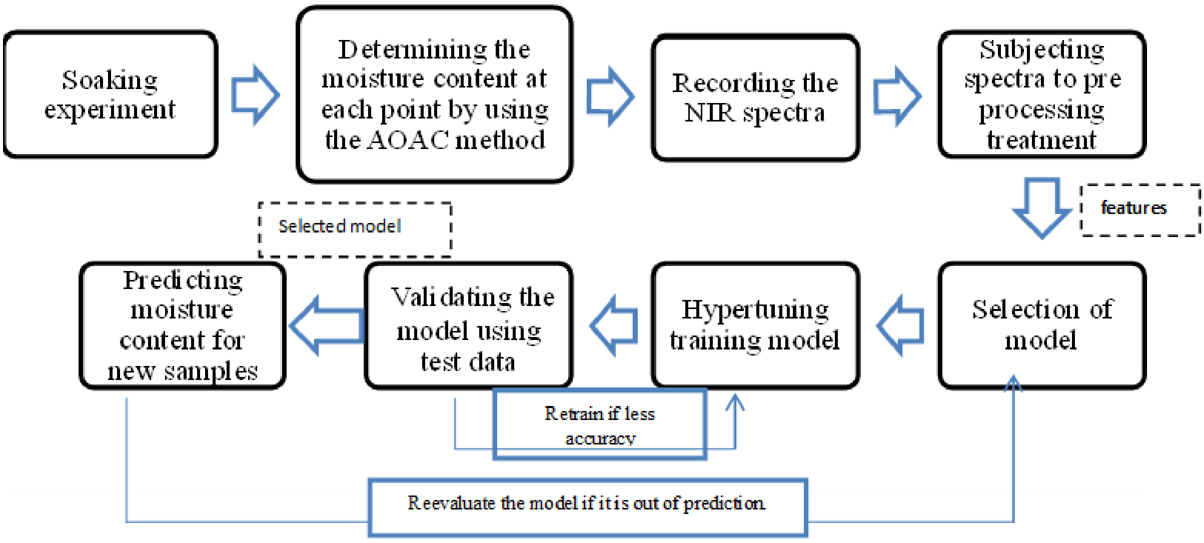


Fig. 3.2.5: Overall methodology for calibrating the NIR spectra for moisture content prediction during soaking

3.2.2.2 Steaming endpoint prediction using spectral data

NIR-based spectra were generated within wavelengths ranging from 740-1070 nm at 5 nm resolution using a portable spectrophotometer (SCiO 1.2, Consumer Physics, Israel) and stored in a cloud system. The spectra were acquired for each experiment mentioned in section 3.2.1.2 by setting the attributes as time and pressure. Around 1 g of samples were taken in the small aperture that comes with the sensor, and the lens-facing head of the sensor was placed over the vessel. Throughout all the experiments, a fixed gap of 1.0 cm was maintained between the NIR sensing device (SCiO 1.2) and the sample with the help of the sample holder. The sensor was connected to the smartphone via Bluetooth and the spectral scans were acquired for each pressure and each temperature in triplicates using the SCiO Lab application on the phone.

The spectral reflectance data for each pressure and each temperature combination, against their degree of gelatinization (DG) value, were arranged in a matrix format. The DG values were taken as labels and the reflectance values at all resolutions were taken as features. The spectral features were pre-processed to avoid noises and gaps. The pre-processing methods used are 1st derivative, standard normal variate, and wavelength selection followed by Savitzky-Golay for smoothing.

The best features (X) matrix and the vector DG values (Y) were split into training, validation, and testing sets (60, 20, and 20% respectively). A total of 140 scans were used in the model development- 84 scans for training, 28 scans for validation, and the rest 28 scans for testing. The training set was employed to develop a PLS-based regression model. The validation set was used for tuning the latent variables of the developed model finding the best fit. The accuracy was tested using statistical metrics R^2 (Eq. 3.54) and RMSE (Eq. 3.55). The predictable accuracy of the trained-validated model was then evaluated using the test data set (Fig. 3.2.6). The performance of the test set was inferred using a cross-validation plot.

$$R^2 = \frac{n\sum_{i=1}^{n} x_{pi}x_{mi} - \sum_{i=1}^{n} x_{pi}x_{mi}}{\sqrt{\left(n\sum_{i=1}^{n} {x_{pi}}^2 - \left(\sum_{i=1}^{n} x_{pi}\right)^2\right)\left(n\sum_{i=1}^{n} {x_{mi}}^2 - \left(\sum_{i=1}^{n} x_{mi}\right)^2\right)}} \tag{3.54}$$

$$RMSE = \sqrt{\frac{\sum_{1}^{n}(x_{pi} - x_{mi})^2}{n-1}} \tag{3.55}$$

Where, x_{pi} is the predicted ith value and x_{mi} is the measured ith value.

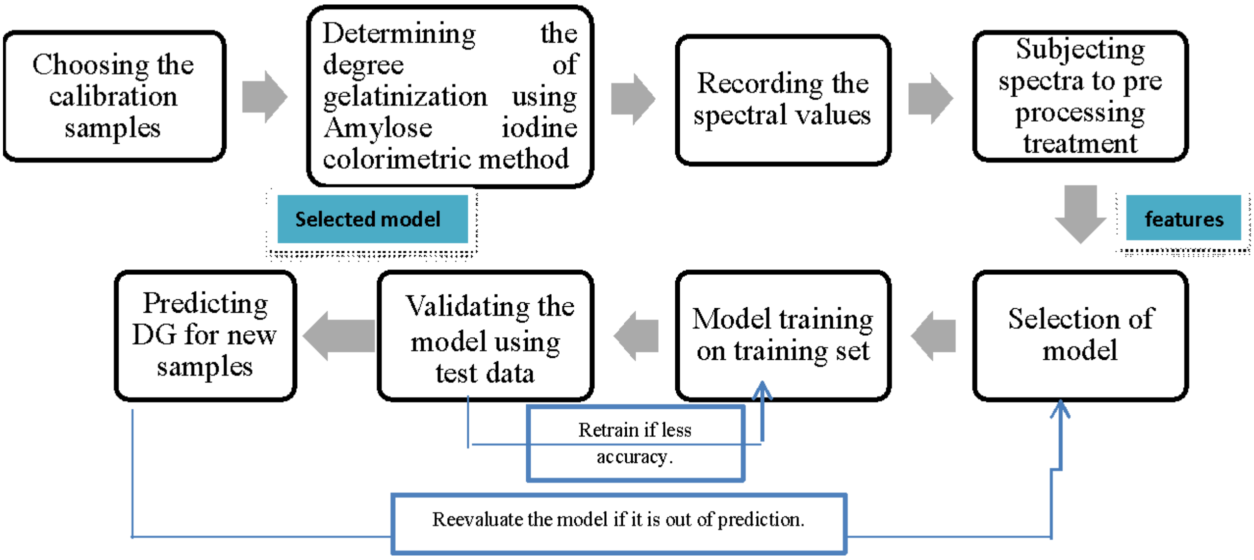
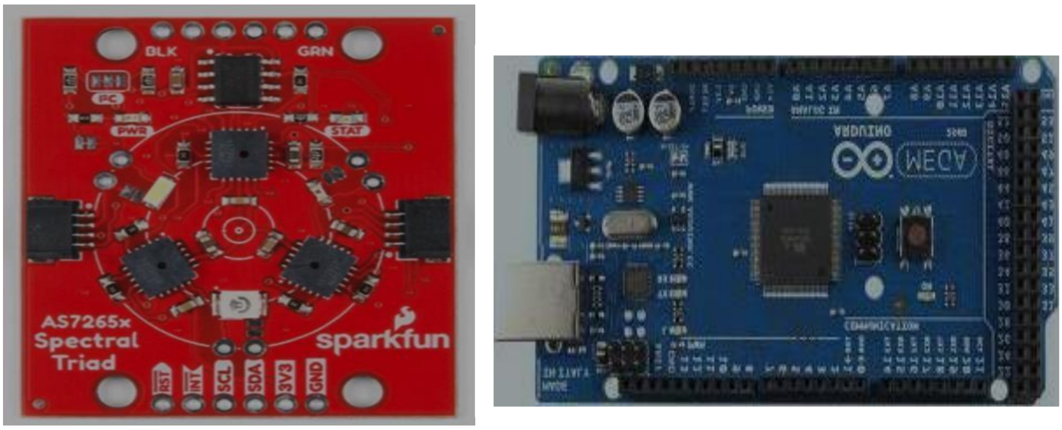


Fig. 3.2.6: Overall methodology for calibrating the NIR spectra for DG prediction during steaming

3.2.2.3 Drying endpoint prediction using spectral data

Spectra were determined both in the visible as well as spectral region using the triad sensor (Model: AS7265x) interfaced with the Arduino Mega board (Model: 2560) (Fig. 3.2.7a, 3.2.7b and 3.2.7c).



a: Triad sensor **b: Arduino Mega board**

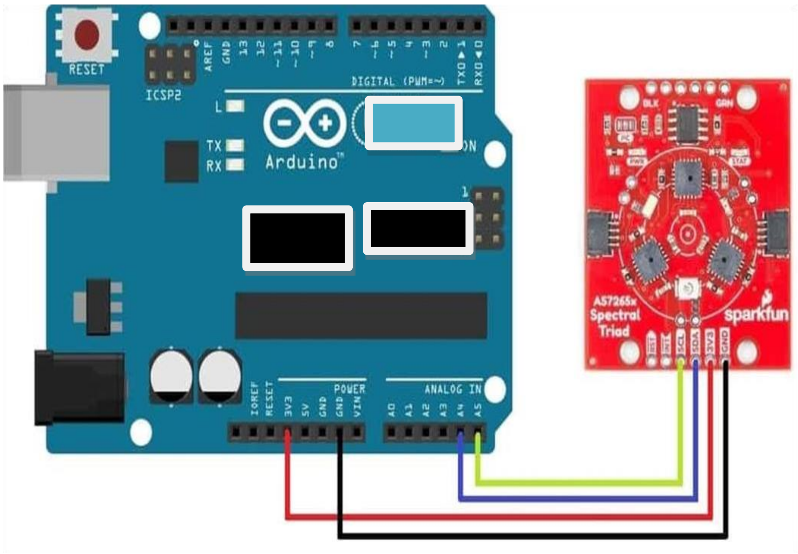


Fig. 3.2.7 Schematic diagram of Triad sensor interfaced with Arduino Mega board

The graphical flow chart was mentioned to give an overview of the methodology for the calibration of spectral data with moisture content during drying. The spectrum of each feature of time and temperatures was obtained in the Putty configuration. De-husked *Komal Chaul* was subjected to

100 min of soaking at 60 °C to achieve 30% moisture content and 12 min of steaming at 0 psi gauge pressure to achieve 99% degree of gelatinization. Later, the samples were put in a tray dryer at 40, 50, and 60 °C, simultaneously the sample spectra were recorded every 10 min for 180 min, giving a dataset of 57x5 scans. In the meantime, respective moisture content was calculated using the gravimetric oven-dried method. The spectra were then labeled into two classes: moisture less than 13% and moisture greater than 13%. ML model procedure of training, validation, and testing was followed with the k-fold validation method. The models that were used were, namely Logistic Regression (LR), Linear Discriminant Analysis (LDA), Classification and Regression Tree (CART), K-Nearest Neighbor (KNN), Naive Bayes (NB), and Support Vector Machine (SVM). Regression analysis for the estimation of moisture from spectral data was done using PLS and ANN.

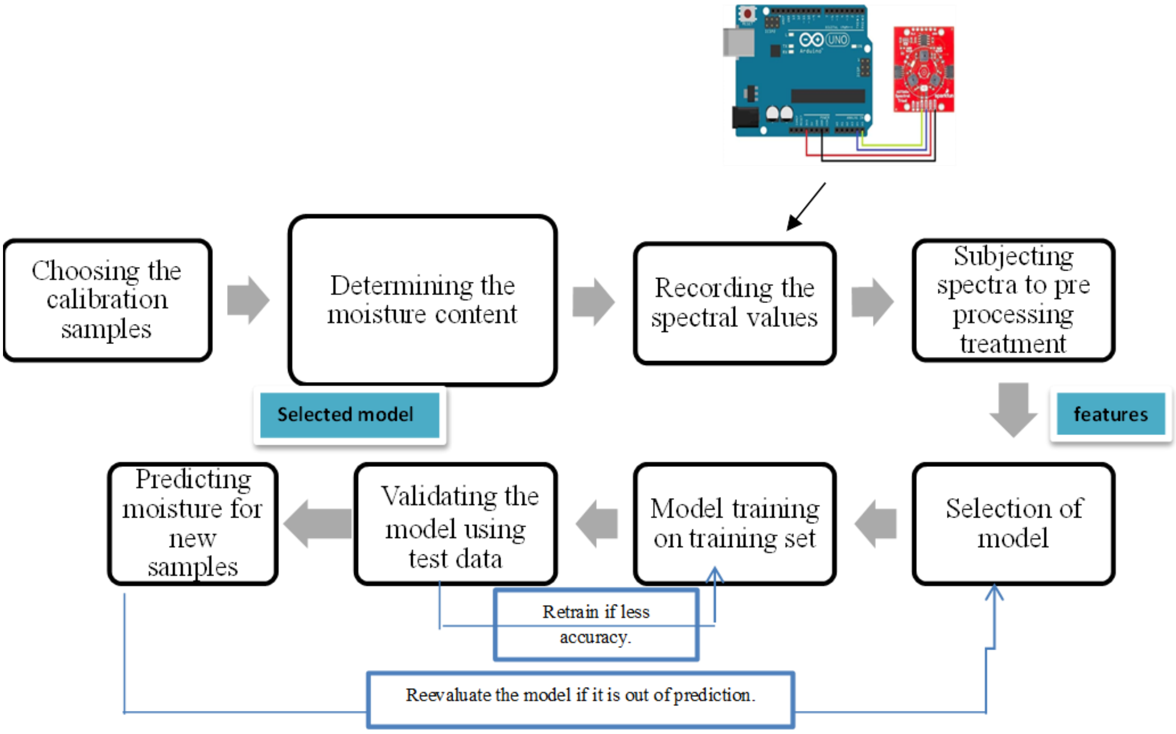


Fig. 3.2.8: Overall methodology for calibrating the NIR spectra for drying process for moisture content calibration

3.2.3 Methodology for studying the physicochemical properties during ageing

A study on the changes of unaged and aged *Chokuwa* rice and parboiled *Chokuwa* rice (*Komal Chaul*) was studied as discussed in the following. Since the softening time is an important property for *Komal Chaul*. A thorough study of cooking time (softening time) estimated throughout the ageing period of a year.

3.2.3.1 Cooking (softening) property

The cooking quality or softening properties of *Komal Chaul* from *Chokuwa* rice of Assam with respect to ageing time was measured and the parameters taken into consideration is the softening time. The time from starting cooking to the first sampling instance at which 90% of kernels are translucent is considered as the cooking time. To calculate the cooking time 10 g of kernels was kept in a hot water bath at 60 ± 5 °C. Eight minutes onwards, at an interval of 1 min, 10 kernels were taken out and pressed between two glass slides. The percentage of translucent kernels was noted, and cooking time was determined when the percentage was above 90% [19].

3.2.3.2 Compositional changes

3.2.3.2.1 Amylose content

Amylose content was measured using the method developed by Juliano (1970) [1], also cited in various other works. For the estimation, 100 mg of ground rice samples were taken, and added 1 ml of distilled ethanol. Then 10 ml of 1 N NaOH was added and was left for 6 h. Afterward, the volume was made to 100 ml in a beaker by adding distilled water. Then taking 2.5 ml of the extract, added about 20 ml of distilled water and then two drops of phenolphthalein reagent. The alkalinity was adjusted by titrating with 0.1 N HCl until the pink color disappeared. 1 ml of iodine reagent was added, and the volume was made to 50 ml and the absorbance values were noted down at 590 nm. Similarly, the standards were prepared at 0.2, 0.4, 0.6, 0.8, and 1 ml of the standard amylose solution; and absorbance values were recorded at the same wavelength. For blank, 1 ml of iodine reagent was added to 50 ml of distilled water. The absorbance values of the standard concentration were plotted against wavelength. The amount of amylose was calculated for the sample using the standard curve.

3.2.3.2.2 Protein content

Crude protein was estimated using the Kjeldahl method which involves digestion-distillation-titration suggested by AOAC (2009) [2]. The protein nitrogen fraction taken into consideration is 5.75.

3.2.3.2.3 Fat content

Crude fat was estimated by the method suggested by AOAC (2009) [2]. Fat was estimated using the Soxhlet method where the sample in a thimble was subjected to extraction method in a petroleum ether solvent, and later followed by the gravimetric method of mass estimation to calculate the percentage of fat. The crude fat percentage can be expressed as (Eq. 3.56),

$$Fat\ (\%) = \frac{(W_f - W_i)}{S} \times 100 \tag{3.56}$$

Where W_f is the weight of a thimble along with extracted fat, W_i is the weight of an empty thimble, and S is the weight of a sample.

3.2.3.2.4 Free fatty content

The storage study is on brown rice, so there is a chance of it getting oxidized due to some amount of bran present in it. As a result, free fatty acid was measured using the protocol suggested by Sadasivam [20]. The free fatty acid is estimated by titrating it with potassium hydroxide (KOH) in the presence of a phenolphthalein indicator. The acid number is defined as the *mg* KOH required in neutralizing the free fatty acid in 1 *g* of sample (Eq. 3.57). The free fatty acid is calculated in terms of oleic acid and the 1ml KOH (0.1 N) is equivalent to 0.028 g of oleic acid.

$$Acid\ value\ (mg/g = \frac{Titre\ value \times Normality\ of\ KOH \times 56.1}{Weight\ of\ the\ sample\ (g)} \tag{3.57}$$

3.2.3.3 Structural, pasting, and textural properties

3.2.3.3.1 X-Ray Diffraction for crystallinity

X-ray diffractometer was used to study the crystallographic structure of our rice sample and to observe the changes in stored rice. Wide-angle X-Ray diffractograms were obtained with diffraction angles, i.e., 2θ of 10-40° around a size of 0.05° with a target Cu Kα value of 1.5 A° (λ) at an operating scan speed of 1 degree per second. The peaks were obtained by plotting and using the '**scipy.signal.find_peaks**' in Google Colab.. The mean percent crystallinity was determined by Eq. 3.57 following Manful et al. [21].

$$\% \, Crystallinity = \left(\frac{\sum Area \ of \ the \ crystalline \ region}{Total \ area}\right) \times 100 \quad (3.58)$$

3.2.3.3.2 Fourier transform Infrared spectroscopy for molecular bond

FTIR was used to measure the molecular changes during the process of ageing. For analysis powdered rice samples were used for aged and unaged rice samples, and the dried rice flour and desiccated KBr powder were thoroughly mixed. Then poured and pressed by using a hydraulic press into a thin pellet. The FTIR spectra were recorded in the range of 4000 to 400 cm^{-1} wavenumber with a resolution of 4 cm^{-1} using an Infrared Spectrophotometer (Model: Frontier IR, Make: Perkin Elmer, USA). The data were plotted using matplotlib library in Python and peaks were found using SciPy library.

3.2.3.3.3 Texture analysis for cooking characteristics

Textural properties of cooked parboiled samples were determined using a texture measuring instrument (TA-HD Plus, Stable Micro Systems, UK). The two-cycle compression test was used with a pre-test and test speed of 0.5 mm/sec, post-test speed of 2 mm/s, strain of 90%, time of 5 s, and trigger force of 0.05 N [22]. The mean values of hardness and stickiness, based on 20 replicated measurements of the same samples were noted.

3.2.3.3.4 Pasting behavior using Rapid Visco Analysis

Rheological properties of samples were measured using the Rapid Visco Analyser Standard 1 measurement profile (ICC Standard Method No. 162) was used. The viscosities were recorded in cP units. The sample (2 g of ground wheat with a particle size of 100 mesh size) was dispersed in distilled water (18 ml) and pre-sheared in an RVA canister at 960 rpm for 10 sec at 50 °C, then at 160 rpm for the remainder of the test. The standard temperature profile stages were holding for 1 min at the initial temperature (50 °C); followed by heating to 95 °C over 3.42 min; and then holding at 95 °C for 2.30 min; afterwards cooling to 50 °C over 3.48 min; and finally hold at 50 °C for 2 min. The total test time was 13 min. The interval between viscosity and temperature readings were 2s (195 data points per RVA curve). Peak Viscosity (PV), Pasting temperature (PT), Holding Viscosity (HV), and Setback Viscosity (SV) were obtained.

3.2.3.4 Temperature-based storage study

A temperature-based storage study was conducted for 6-month-old *Komal Chaul* for a storage period of 2 months based on a study suggesting that the period from 6 to 8 months shows maximum changes in cooking and pasting behavior. Changes in rehydration ratio or water uptake ratio and pasting behavior were studied at an interval of 3 days for two months for *Komal Chaul* stored at 37 °C and 4 °C. The thermal properties and surface morphology were also determined at the end of a year of those samples.

3.2.3.4.1 Water uptake behavior

The water uptake ratio was measured using the gravimetric method of moisture determination (Eq. 3.23). The rehydration ratio was measured after soaking at 60 °C for 20 min taking 10 g of sample and 20 ml of water [23]. The water uptake ratio was measured using Eq. 3.59.

$$Water\ uptake\ ratio = \frac{Mass\ after\ rehydration}{Initial\ mass} \qquad (3.59)$$

3.2.3.4.2 Pasting behavior using Rapid Visco analyzer

Rapid visco analysis was performed in a rheometer (Make: Anton Paar, Model: MCR 72). The pre-shear measurement for mixing was 960 rpm for 10 s at 50 °C. The rapid visco analysis was performed at 160 rpm and the temperature cycle started at 50 °C for 50 s, then heating from 50 °C to 95 °C for 450 s and holding at 95 °C for 300s followed by cooling for 450 s and holding at 50 °C for 120 s. The time interval was 2 s. The interval between viscosity and temperature readings was 2 s. Peak Viscosity (PV), Pasting temperature (PT), Holding Viscosity (HV) and Setback Viscosity (SV) were obtained.

3.2.3.4.3 Differential Scanning Calorimetry for the thermal properties

The thermal properties of rice stored at 4 °C and 37 °C were compared. The differences in the thermal properties of aged and unaged rice were determined using Differential Scanning Calorimetry (Model: JSM-6360, Make: Perkin Elmer, USA). Initially, 10 mg of rice in 30 µl of distilled water was put in the sample pan sealed and was kept overnight. Next day, the pan was then kept in the DSC heating chamber and the temperature was increased from 30 °C to 100 °C at a rate of 1 °C/min. The onset gelatinization temperature (T_g), peak temperature (T_p), and conclusion temperature (T_c) were noted down along with gelatinization enthalpy [24].

3.2.3.4.4 Surface Morphology using Scanning Electron Microscope (SEM)

The changes in the rice grain structure for aged and unaged using a scanning electron microscope (Model: JSM-6390LV, Make: Jeol, Japan). The samples went through a process of plunging into nitrogen gas, then sublimating off the ice, and then coating with pure platinum. Afterward, the coated samples were visualized at -180 °C on a cold stage of the SEM at a resolution of 500 µm and 1000 µm.

3.2.4 Methodology for prediction of ageing time

Komal Chaul (parboiled *Chokuwa* rice) obtained from freshly harvested *Chokuwa* paddy is taken for ageing study. The rice was stored in LDPE pouches and those packets were kept in a closed box at room temperature. The temperature variation throughout the year was 37-23 °C. The target parameter here was the time of storage (degree of ageing) associated with quality attributes during ageing. The respective spectrum of samples was taken over a period of one year on a weekly basis. The procedure involves:

- Firstly, created a NIR-based spectral library of aged and unaged rice samples with their respective storage time. The storage time was considered as the reference parameter or target parameter. The respective spectra were acquired in the range of 740-1050 nm using the NIR sensing device (SCiO 1.2) (Fig. 3.4).
- Initially, the whole spectrum was taken as a reference spectrum with a resolution of 5 nm giving us 63 points for every spectrum. The dataset was divided into training, validation, and test sets.
- Preprocessing of spectra is an essential step to enhance its quality, remove noise, and normalize the data for further analysis in regression.
- Developing an ML-based predictive model for estimating the time of ageing of *Komal Chaul* using the spectral library. The PLS process is explained in figure 3.4.1.
- The preprocessed data was then trained on PLS, the PLS algorithm iteratively calculated a set of latent variables over 40 numbers of components. The optimal number of components was chosen based on the Mean squared error.
- That model was then validated using statistical metrics: Regression coefficient (R^2), Relative Error percentage (REP), and Root mean squared error (RMSE) (Eq. 3.60-3.62).

$$R^2 = \frac{n\sum_{i=1}^{n} x_{pi}x_{mi} - \sum_{i=1}^{n} x_{pi}x_{mi}}{\sqrt{\left(n\sum_{i=1}^{n} {x_{pi}}^2 - \left(\sum_{i=1}^{n} x_{pi}\right)^2\right)\left(n\sum_{i=1}^{n} {x_{mi}}^2 - \left(\sum_{i=1}^{n} x_{mi}\right)^2\right)}} \quad (3.60)$$

$$RMSE = \sqrt{\frac{\sum_{1}^{n}(x_{pi} - x_{mi})^2}{n-1}} \tag{3.61}$$

$$REP = \frac{x_{pi} - x_{mi}}{x_{mi}} \times 100 \tag{3.62}$$

Where x_{pi} is the predicted ith value and x_{mi} is the measured ith value.

- The validated model was again used for prediction.
- There are specific regions in the spectrum that contributed more to the PLS regressor loading values, therefore a coefficient weightage-based selection of optimal features was done to get the wavelengths more distinctively contributing to the PLS regression of time calculation.

The next step involved the classification of spectral data based on their ageing time. The spectral database was coded according to 1-month classification consisting of 12 classes, 3 months of classification and 6 months of classification. The dataset was divided into training (70%), validation (20%), and testing (10%). The dataset was then trained, validated, and tested on classification models: LDA, KNN, CART, NB, SVM, and RF. The performances of the classification were measured using two metrics: classification accuracy (Eq.3.63) and F1_score (Eq. 3.64).

$$Accuracy = \frac{Number\ of\ correctly\ classified\ sample}{Total\ number\ of\ samples} \tag{3.63}$$

$$F1\_score = \frac{2 \times precision \times recall}{precision + recall} \tag{3.64}$$

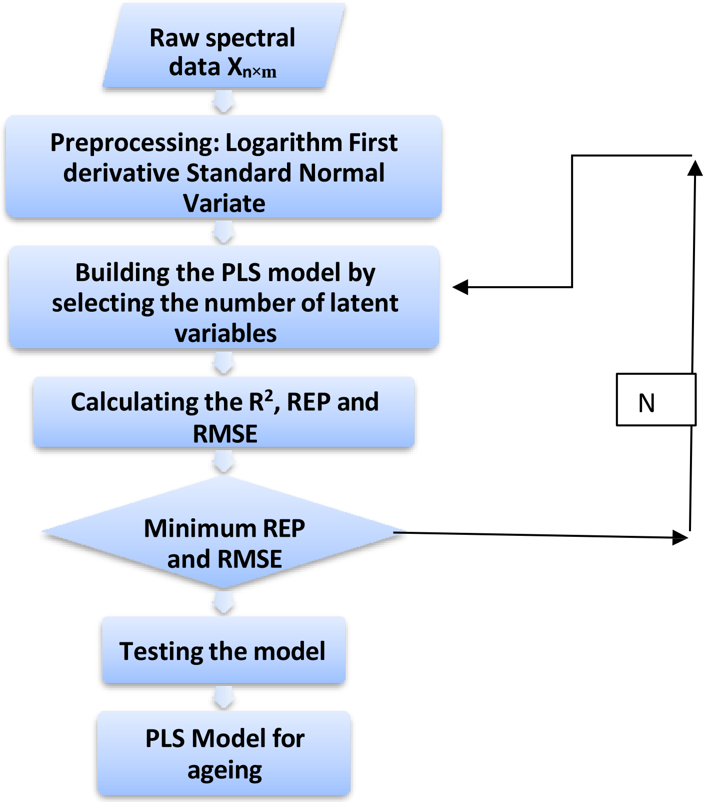


Fig. 3.4.1: Flowchart of a PLS process

3.3 Summary of Chapter III

The first objective was a study of the kinetics of the parboiling process of *Chokuwa* rice for the manufacturing of *Komal Chaul*. The methods involved in the first objective are applications of Peleg model, Midili Kucuk and Fick's diffusion equation, the application of first-order kinetics for studying the gelatinization process during steaming followed by moisture diffusion during steaming; lastly, the drying kinetics was studied using semi-empirical models and a mechanistic approach using Fick's law. The second objective was to develop an ML-based model for which regression models like PCR, PLS, and ANN were used, and classification models like LR, LDA, KNN, NB, CART, SVM, and RF were used. The third objective was to study storage and how it affects physicochemical characterization like pasting behavior using RVA, thermal properties using DSC, molecular bond using FTIR, surface structure using SEM along with throughout cooking parameters for characterization of aged and unaged *Komal Chaul*. Lastly, the methodology implemented for objective 4 was the ML-based regression technique PLS to relate ageing time with spectral data and ML-based classification of ageing time.

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