Chapter 3

Objective 1 has been discussed under this chapter. It is divided into three sub-chapters (3A, 3B and 3C). Based on the comparison of the physical and chemical qualities of head rice and broken rice flours, the first sub-chapter discusses the study for the selection of broken rice fraction for the production of rice noodles. The second sub-chapter discusses the usability of medium broken rice in rice noodle production and identifies the suitable particle size for better rice noodle quality. The third sub-chapter deals with the modification of rice starch isolated from medium broken rice and finding the optimal process conditions of the modification processes.

Chapter 3A: Comparative analysis of chemical composition, functional, morphological and pasting characteristics of head rice and broken rice

3A.1. Introduction

Rice milling industry is the largest agro-based industry in India [39]. In India, rice milling is mostly carried out in small and medium size rice mills generating around 60-68% head rice with 10-25% brokens and in relatively a few modern type mills that generates 68-72% head rice recovery with 5-7% brokens [83] indicating a considerable loss in head rice yield. Kernel size less than 75% are considered as brokens [14], which are regarded as a low value by-product of the rice milling process. It is believed that broken rice is the result of internal tensions and strains brought on by moisture sorption, which create fissures and cracks that result in grain breakage during milling [117]. Moreover, rice grains are not uniform in terms of composition and properties throughout their length. Due to this reason, the rice brokens generated from different positions of the whole rice grain are most likely to possess different sizes and thereby have dissimilar properties. In addition, the degree of milling affects the composition of the whole grains and brokens. Therefore, the functionality of rice flour obtained from rice brokens will primarily depend on the size of the broken rice. According to literature, rice brokens differ from head rice in terms of their functional characteristics; in particular, they exhibit softer gel and lower paste viscosities [117], which decline with reduction in size of the brokens. Hence, separating the brokens on the basis of size will be effective to investigate the ultimate properties of their flour that will eventually help to identify their end use [79].

The commercial use of its by-products is a key factor in the economics of the rice milling industry [76]. In the rice industry, broken rice is considered as low-value waste that has been still largely underutilized [2, 86], thus need ways to utilize it [122]. However, because of their cost-effective applicability in a variety of items such as, gluten free foods [87, 111, 113], baby foods [82], rice breads [37], noodles [2, 24], fermented rice products [66, 80], pet foods [59, 110], and biodegradable films [119], edible films [95] and edible cutlery [79] The demand of broken rice has risen in the past few years. Broken rice may serve as an important source of raw material that could be used as an ingredient in various processed foods to improve food security in the country [76]. Usage of broken rice to produce rice flour and starches as an alternative to head rice will be most justifiable.

It is therefore, hypothesized that the different fractions of broken rice obtained from a single variety of rice will vary in size and chemical composition, and consequently produce rice flour of different functional properties. Thus, the goal of this study was to fractionate the rice brokens on the basis of size and determine the chemical composition, functional and pasting properties in addition to gel hardness and morphology of the whole and broken grains in order to identify its possibility to be used in rice noodle production.

3A.2. Materials and methods

3A.2.1. Materials

A preliminary analysis of the amylose content, pasting properties and gel hardness of four commonly cultivated and consumed rice varieties viz. Ranjit, Parimol, Pankaj and Aijong (whole rice was used) were analysed. Ranjit variety showed the highest amylose content and higher pasting properties and gel hardness (data not shown) than the other varieties. Thus, *Ranjit* variety as a source of head rice and broken rice was selected for the present study.

Paddy of *Ranjit* variety was supplied by a farmer from Kokrajhar District, BTR, Assam, which was then milled in a local small rice mill. Head rice and brokens were separated manually using traditional household commodity (*Dala* in Bodo dialect). After being cleaned to remove any foreign objects, the head rice and broken rice were packed separately in plastic bags and kept in the refrigerator (4 ^{o}C).

3A.2.2. Physical characteristics of head rice (HR)

To determine the physical properties of the HR of *Ranjit* variety, mean length and mean width of 10 grains were analysed using a vernier calliper. The length to width ratio was calculated thereafter. Grain length and shape (length to width) was determined as per the classification given by Juliano et al. [55].

3A.2.3. Chemicals and reagents

All the chemicals and reagents used for various analyses in the present research work were of analytical grade and were purchased from Merck, SRL, Himedia, and Sigma India.

3A.2.4. Classification of broken rice

A sieve analysis was performed to segregate the broken rice into small, medium and large sizes as per the method as mentioned in [79]. For this, three laboratory sieves with standard ASTM no. 10 (2 mm), 12 (1.7 mm) and 20 (0.85 mm) were fitted in a sieve shaker (Aimil, India) and operated at 150 rpm for 15 min. The brokens that were retained on 2 mm sieve were classified as large brokens (LBs), those retained on 1.7 mm sieve after passing through 2 mm sieve were classified as medium brokens (MBs), and those retained on 0.85 mm after passing through 1.7 mm sieve were classified as small brokens (SBs).

3A.2.5. Preparation of rice flours

The brokens and whole grains were manually cleaned, washed with tap water, air dried for 1 h and ground into flour using a mixer grinder (Philips, model HL7505/00) and sieved through 0.425 mm sieve. Rice flour was dried at 40 ^{o}C for 24 h in an oven (Optics Technology, Delhi). The rice flours were sealed in airtight containers and stored in the refrigerator (4 ^{o}C).

3A.2.6. Chemical composition of rice flours

3A.2.6.1. Moisture content

Moisture content of flours were analysed by moisture analyser (Sartorius MA35, Germany) according to the standard protocol and inbuilt profile (110 ^{o}C).

3A.2.6.2. Protein content

Protein content was determined using an automated Kjeldahl unit (KjelFlex K-360 by Buchi, Switzerland) according to the standard Association of Official Analytical Chemists (AOAC) method [7]. Briefly, 2 g rice flour was digested with 10 ml concentrated sulphuric acid added with copper sulphate and potassium sulphate. Digestion was continued at 350 ^{o}C until the content becomes clear and a bluish green colour developed. The cooled digested sample was distilled using 40% sodium hydroxide solution and the ammonium gas generated during distillation was passed over boric acid solution in a conical flask. Distillate was thereafter titrated with 0.1 N HCl using methyl red as indicator. The nitrogen content of the sample was quantified using the Eq. 3A.1 and protein content was calculated using Eq. 3A.2.

$$Nitrogen\ content\ (\%, db) = \frac{Normality\ of\ HCl \times titre\ volume\ of\ acid \times 14 \times 100}{Weight\ of\ the\ sample \times 1000} \quad \text{Eq. (3A.1)}$$

$$Protein\ content\ (\%, db) = Nitrogen\ \times 6.25 \quad \text{Eq. (3A.2)}$$

3A.2.6.3. Fat content

Fat content was analysed using a Soxhlet apparatus according to the standard AOAC method [7]. Previously dried 2 g rice flour was taken in a pre-dried cellulose thimble, placed in a pre-weight extraction flask and extraction was carried out at 100 ^{o}C for 2 h using petroleum ether (boiling point range of 60-80 ^{o}C) in a SOCS PLUS Pelican Equipment. After extraction, the residual solvent was evaporated at 200 ^{o}C and collected by condensation. The flask was dried at 70 ^{o}C for 30 min to remove residual moisture, kept in desiccator until cooled and weight was taken. Fat content was determined by the following formula,

$$Fat\ content\ (\%, db) = \frac{Weight\ of\ the\ flask\ and\ fat - weight\ of\ the\ empty\ flask}{Dry\ weight\ of\ the\ rice\ flour} \times 100 \quad \text{Eq. (3A.3)}$$

3A.2.6.4. Ash content

Ash content was estimated in a muffle furnace as per the standard AOAC method [7]. Weight of the empty dry crucible was taken followed by measurement of 2 g of rice flour (rice flour after moisture content determination). The crucible kept in the muffle furnace at 600 ^{o}C for 6 h. After 6 h, the sample containing crucible was allowed to cool in desiccator and weighed thereafter. Ash content was estimated by the formula given in Eq. (3A.4)

$$Ash\ content\ (\%, db) = \frac{Final\ weigt - weight\ of\ the\ crucible}{Initial\ weight\ of\ the\ flour} \times 100 \qquad \text{Eq. (3A.4)}$$

Where, final weight refers to weight of ash and the crucible weight.

3A.2.6.5. Starch content

Total starch content in the sample was estimated by anthrone method [96]. Briefly, 0.5 g of the sample was homogenised with hot ethanol (80%) and washed repeatedly to remove the free sugars. When the supernatant did not turn green with anthrone reagent, the washing was stopped. The residue was dried and mixed with 5 ml distilled water, 6.5 ml 52% perchloric acid, and kept at 0 ^{o}C for 20 min (for starch extraction). Supernatant was obtained by centrifugation at 3000 rpm for 10 min. Extraction was repeated with fresh perchloric acid, centrifuged to collect the supernatant until the volume was made up to 100 ml. In a test tube, 0.2 ml of supernatant was placed and the volume was adjusted to 1 ml with distilled water. For standard curve, stock solution was made by dissolving 100 mg D-glucose (Sigma-Aldrich) in 100 ml distilled water. Distilled water was used to dilute a 10 ml stock solution to 100 ml (working standard). A series of different concentration of working standards were taken in test tubes (0.2, 0.4, 06, 0.8 and 1 ml) and volume adjusted to 1 ml with distilled water (except in 1 ml working standard). To each test tube, 4 ml anthrone reagent was added and incubated for 8 min in a boiling water bath. After cooling, the colour intensity was read at 630 nm in a spectrophotometer (Perkin Elmer, UV/VIS spectrometer, lambda 35). A standard curve was prepared by using the colour intensity readings of the standard glucose. The standard curve was used to estimate the sugar concentration of the rice flour samples and the starch content was obtained by multiplying with 0.9 as shown in Eq. (3A.5) given below,

$$Total\ starch\ (\%, db) = Percentageof\ glucose\ per\ dry\ sample\ weight \times 0.9 \qquad \text{Eq. (3A.5)}$$

3A.2.6.6. Amylose content

Amylose content was determined by the method described elsewhere [108]. Rice flour was defatted with methanol for about 18 h and air dried at ambient temperature. Then, 100 mg of defatted rice flour was taken in 100 ml Duran glass bottles and 1 ml absolute ethanol was added to wet the flour followed by 10 ml of 1 N NaOH that was added gently by the wall of the bottle. The wetted flour solution was left overnight (approx. 18 h) and then heated in a water bath at 100 ^{o}C for 2-3 min. The volume was

adjusted to 100 ml with distilled water. A solution of 100 ml standard amylose was prepared in the same way as described above for rice flour. For neutralization and colour development, 5 ml of the rice flour dispersion or 1 ml of standard amylose solution was taken in a test tube, 2-3 drops of phenolphthalein and 50 ml distilled water were added and neutralized with 1 N and 0.1 N HCl. To this, 2 ml of freshly prepared iodine reagent was added, volume adjusted to 100 ml by adding boiled and cooled distilled water. For the blank, 2 ml iodine solution was taken and volume made up to 100 ml. After that, all the samples were kept for 30 min in darkness and the developed colour was read in a spectrophotometer (Perkin Elmer, UV/VIS spectrometer, lambda 35) at 630 nm. Amylose content was calculated using the Eq. (3A.6) as follows:

$$Amylos\ content\ (\%, db) = \frac{R}{A} \times \frac{a}{r} \times \frac{1}{5} \times 100 \qquad \text{Eq. (3A.6)}$$

Where, R= Reading of rice flour dispersion, A =Reading of standard amylose solution. a =Amount of standard amylose weighed (mg), r =Amount of rice flour weighed (mg).

3A.2.7. Functional properties of rice flours

3A.2.7.1. Loose bulk density (LBD) and packed bulk density (PBD)

Packed bulk density is the highest achievable density of the material that can be attained with compression. The method outlined by Falade et al. [33] was used to determine the bulk density of both loose and packed rice flours. The weight of a 10 ml graduated glass cylinder filled with rice flour sample up to the 10 ml mark was measured. The measuring cylinder was tapped (50 times) until the volume of the flour did not change. The weight and volume of flour filled cylinder after tapping were recorded. Loose density and packed bulk density were calculated by the formula given in the Eq. (3A.7) as the ratio of bulk weight to volume before and after tapping respectively and expressed in g/ml

$$Bulk\ density\ (\ g/ml) = \frac{Weight\ of\ sample}{Volume\ of sample} \qquad \text{Eq. (3A.7)}$$

3A.2.7.2. Water absorption capacity (WAC) and oil absorption capacity (OAC)

WAC and OAC were determined as per the method described by Falade et al. [33]. In a 50 ml pre-weighed centrifuge tube, 2.5 g rice flour was dispersed in 30 ml distilled water. The tubes were immersed in a 30 ^{o}C water bath and agitated occasionally for 30 min. For 10 minutes, the suspension was centrifuged at 3500 rpm. After

discarding the supernatant, the sediment's weight was measured. WAC was calculated as given below:

$$WAC\ (g/ml) = \frac{Weight\ of\ the\ sediment}{Weight\ of\ dry\ sample} \quad \text{Eq. (3A.8)}$$

A pre-weighed, clean, dry centrifuge tube was filled with 1 g of rice flour, and the weight was recorded. The tube was filled with 10 ml of refined soybean oil, which was then well mixed with a glass rod. For 20 minutes, the tube's contents were centrifuged at 3500 rpm. After the supernatant was taken out, the tube was weighed. The OAC was determined as weight gain and expressed as bound oil in g/ml.

3A.2.7.3. Swelling power (SP) and solubility (SOL)

A 50 ml centrifuge tube was filled with 15 ml of distilled water and then 0.5 g of flour was added and thoroughly mixed. To gelatinize the starch present in flour, the slurry was heated at 92 ± 2 °C for 30 min in a shaking water bath. The centrifuge tube was cooled to ambient temperature and centrifuged at 3500 rpm for 15 min. A Petri dish was filled with the supernatant, which was then dried at 100 °C until the weight was constant. The weight of the wet sediment in the centrifuge tube was measured. Each flour sample was subjected to analysis in triplicate. The following equations were used to calculate swelling power and solubility percentage [49].

$$Swelling\ power\ (g/g) = \frac{Weight\ of\ the\ wet\ sediment}{Weight\ of\ initial\ dry\ sample} \quad \text{Eq. (3A.9)}$$

$$Solubility\ (\%) = \frac{Weight\ of\ dried\ solid\ supernatant}{Weight\ of\ initial\ dry\ sample} \times 100 \quad \text{Eq. (3A.10)}$$

3A.2.8. Pasting properties and gel hardness

Pasting characteristics of rice flours were determined by a Rapid Visco Analyzer (RVA Starchmaster 2, Perten Instruments, Sweden) [49]. Briefly, 3 g of rice flour was weighed and mixed with 25 ml of distilled water in the RVA canister. The procedure follows a heating and cooling cycle for 13 min from 50 °C to 95 °C then cooling back to 50 °C. For the whole the procedure, the canister was rotated at a speed of 160 rpm. Pasting temperature (PT), peak viscosity (PV), hot-paste viscosity (HPV), breakdown (BD), setback viscosity (SB), and final viscosity (FV) were recorded. BD can be calculated as BD = PV - HPV whereas SB can be calculated as SB = FV - HPV.

The gelatinized paste in the canister was kept overnight at room temperature after the RVA test in order to promote gel formation. To avoid moisture loss during storage, the canister was properly wrapped in parafilm. The gel's height was 20.0 mm, and its

diameter was 37.0 mm. Using a texture analyser, the hardness or texture of the gel was assessed (TA.XT plus, Stable Micro System Ltd, United Kingdom) [49]. The gels were punctured at 1.0 mm/s using a stainless-steel probe (P/6, 6.0 mm diameter) to 10.0 mm distance. The positive peak force in the graph was reported as gel hardness (GH).

3A.2.9. Scanning electron microscopy (SEM)

Surface morphology, transverse section and flour of the head rice, medium broken rice and small broken rice were analysed under SEM (JSM 6390LV, JEOL, JAPAN). For transverse section and surface morphology, the rice samples were carefully dissected with a sharp blade. Sample was fixed on SEM stub with carbon adhesive tape and coated with platinum. SEM was operated at 20 kV acceleration voltage and image were taken under magnifications of 40X, 200X, 2000X, and 5000X at 500, 100, 10 and 5 µm scale respectively.

3A.2.10. Statistical analysis

Unless otherwise specified, all analyses were performed in several replicates, with the mean value presented. Duncan's multiple range test (DMRT) was used in the SPSS (version) to examine significant differences between the mean values at a significance level of $p < 0.05$.

3A.3. Results and discussion

3A.3.1. Physical properties of HR

The mean length and width of the HR of *Ranjit* variety is 5.53 ± 0.34 mm and 2.10 ± 0.07 mm respectively. Thus, *Ranjit* is a medium length grain as the average length is in between 5.51-6.6 mm. The length-width ratio is 2.63 ± 0.16 mm indicating that the grain shape is medium as per the classification given by Juliano [55].

3A.3.2. Classification of broken rice

The sieve analysis of broken rice yielded two fractions of broken rice namely MBs and SBs. The amount of rice brokens retained on 2 mm sieve was zero; indicating that no LBs were present in the whole batch. The fraction of medium and small broken rice was estimated to be 57.55 ± 4.91% and 40.49 ± 4.65%, respectively. This result showed that *Ranjit* rice variety generates brokens that are smaller than 2 mm in size which is relatable because *Ranjit* is a medium length and medium shaped variety of rice. Moreover, the MBs was present in higher quantity than SBs. Researchers have reported

higher amount of medium brokens as well [17, 79]

3A.3.3. Chemical composition

The results of chemical composition are listed in Table 3A.1. Moisture content of rice flours was in the range of 7.53-7.79 (%, db). The values are relatively lower than the reported literature values [19, 32]. SBs flour registered the highest protein content that was significantly higher than HR and MBs. Higher protein content in small brokens than whole grain, large and medium brokens were reported [15-16]. There are numerous potential causes for differences in the protein composition of brokens of different sizes. First, difference in the composition of rice grain across its length, the tips and the middle portion in particular [79] that generates brokens with different chemical composition. Second, when compared to the grain centre, the architecture of the rice grain periphery is very different. Compared to the central cells, the peripheral cells have smaller diameters and more protein [85]. Third, thinner kernels have been reported to contain higher protein content, incompletely filled with starch during development and more vulnerable to developing varied degrees of brokens during milling [40]. Fourth, it was observed that some fractions of SBs were very thin and had slightly coloured outer cover (which might be due to the presence of unremoved bran). It is also plausible that some fractions of SBs produced were undermilled which broke during milling and lacked proper polishing. Consequently, it contained relatively higher protein content, since bran contains higher level of protein than the starchy endosperm [54].

Fat content of SBs flour was higher than MBs ($p \leq 0.05$) and HR flour. Fats within the endosperm are distributed unevenly, with the outer layer having the highest concentration and the central portion having the lowest. During milling most of the fat content is removed as bran and polish. High fat content of SBs and MBs may be attributed to the presence of some bran [77]. The ratio of thick-thin kernels affects the surface lipid content of a batch of milled rice [22-23]. Thick grains possess low fat content for a given degree of milling [21]. This is attributed to easier bran removal in thick grains than the thin grains because thin grans are more prone to breakage during milling. The presence of some thin immature broken kernel was evident in the SBs used in the present study that could be the reason for its high fat content.

Table 3A.1 Chemical composition of SBs, MBs and HR flours.

Types of flour	Moisture (%, db)	Protein (%, db)	Fat (%, db)	Ash (%, db)	Total starch (%, db)	Amylose (%, db)
HR	7.53± 0.23[a]	6.18 ± 0.53[ab]	1.05 ± 0.12[a]	0.32 ± 0.06[a]	84.73 ± 0.87[b]	25.77 ± 0.35[b]
MBs	7.89 ± 0.27[a]	6.12 ± 0.17[a]	1.27 ± 0.24[a]	0.30 ± 0.04[a]	82.05 ± 1.21[a]	24.95 ± 0.51[ab]
SBs	7.79 ± 0.41[a]	6.76 ± 0.29[b]	1.35 ± 0.16 [b]	0.36 ± 0.05[a]	79.62 ± 1.43[a]	24.37 ± 0.86[a]

Results are represented as mean ± SD; Significant differences ($p \leq 0.05$) exist between the mean values with different superscript in a column.

Table 3A.2 Loose and packed bulk density, water and oil absorption capacity of SBs, MBs and HR flours.

Types of flour	Loose Bulk Density (g/ml)	Packed Bulk Density (g/ml)	Water absorption capacity (g/ml)	Oil absorption capacity (g/ml)
HR	0.60 ± 0.02[b]	0.91 ± 0.01[a]	2.20 ± 0.001[c]	2.23 ± 0.02[ab]
MBs	0.57 ± 0.02[ab]	0.88 ± 0.02[a]	2.11 ± 0.002[b]	2.20 ± 0.02[a]
SBs	0.56 ± 0.01[a]	0.89 ± 0.01[a]	2.04 ± 0.003[a]	2.25 ± 0.01[b]

Results are represented as mean ± SD; Significant differences ($p \leq 0.05$) exist between the mean values with different superscript in a column.

3A.3.4. Functional properties

Bulk density, and water and oil absorption capacity of SBs, MBs and HR flours are presented in Table 3A.2. A product's bulk density determines its packing characteristics, including weight, handling requirements, and the kind and suitable type of package for food storage and their transportation. The loose bulk density results showed that SBs and HR had significantly different values. However, no statistical difference in the packed bulk density of the flours was observed; thereby they may have similar packing characteristics. WAC denotes the capacity of the flour to react with limited amount of water. HR showed significantly high WAC whereas SBs had significantly lower WAC. Higher WAC of HR may be attributed to the presence of higher starch content and low protein content since the hydrophilic components of flours, such as charged side chains of proteins and carbohydrates influence WAC [64]. The OAC it contributes significantly to the mouthfeel and flavour retention of the product. The results reveal that the OAC of SBs was significantly higher than MBs and HR. Protein is the most important chemical component influencing oil absorption capacity. SBs also contained highest protein among the flour samples. Non-polar side chains of amino acid can interact hydrophobically with lipid. Higher OAC of SBs is ascribed to the higher protein content, resulting in more hydrophobic associations between protein and fat in the flour [53].

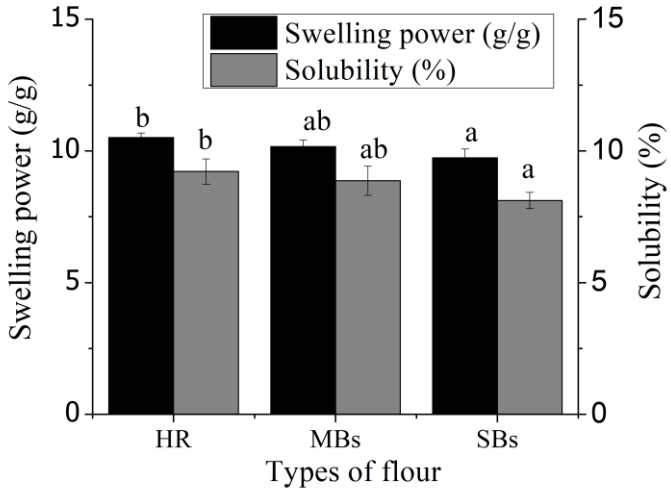

Fig. 3A.1 Swelling power and solubility of SBs, MBs and HR flours.

The swelling power of SBs flour was the lowest and HR flours was the highest (Fig. 3A.1.). The values are in line with previously reported results of rice flours [35]. Proteins being closely packed with the rice starch amyloplast in some rice cultivars

cause a delay in water absorption into the starch granules, which reduces swelling power. Further, the phospholipids present in the starch may react with amylose when heated, give pressure on the amylopectin thus preventing water binding and lowering of swelling power. Thus, protein and fats are negatively correlated to the water uptake that could be the reason of lower swelling power in SBs. Ogawa et al. [85] also revealed that proteins and lipids affect water permeation during cooking of rice. The structural architecture of amylose and amylopectin affects the swelling power of rice flour, with the former inhibiting granular swelling [35]. Hydrogen bonds aid in strengthening the lateral chains of amylopectin and the double helices of amylose. When the starch is hydrated and heated, the hydrogen bonds are broken and replaced with water [26]. The high amylopectin content, on the other hand, contributes to both a greater swelling capacity [107]. It was evident that HR contained higher starch content than MBs and SBs (Table 3A.1) and thus presence of relatively higher amylose and amylopectin than SBs can be assumed that may be one of the reasons behind high swelling power of HR than SBs.

SBs flour showed the lowest solubility ($p \leq 0.05$) followed by MBs and HR (Fig. 3A.1.). Solubility is affected by the amount and size of amylose and amylopectin present in the starch [42]. Low solubility in SBs may be ascribed to the marginally low amylose content which is negatively correlated to solubility. Amylose is mostly present at the central part of starch granule so there will be restricted amylose leaching, thus lowering the solubility [35, 126]. Furthermore, lipid can form complexes with amylose and, to a lesser extent with amylopectin upon heating. This complex prevents starch swelling, amylose exudation during gelatinisation, and thereby lower the amount of soluble amylose [105]. This stands plausible for the low solubility of the SBs since it has higher fat content than MBs and HR (Table 3A.1).

3A.3.5. Pasting properties and gel hardness

The results presented in Fig. 3A.2. and Table 3A.3 showed that HR had higher paste viscosities than the broken fractions. Interestingly, the pasting properties decreased with decreasing size of brokens; SBs showed lower pasting properties than MBs. PT provides an indication of the minimum temperature required to cook the flour [99]. SBs had significantly higher PT than HR (Table 3A.3). This results positively correlates with the results of chemical composition (Table 3A.1) and functional properties (Table 3A.2 and Fig. 3A.1) of the flours. The most plausible reason behind high PT of SBs underlies

in the formation of amylose-lipid complex during heating that resists water permeation into the starch and their swelling, thereby requires higher temperature to disrupt the complex [29]. PV of HR and MBs were higher than SBs that corresponds to higher swelling capacity of starch in former two flours and lower swelling capacity of starch in the latter. BD of flour serves as a measure of its capacity to withstand heat and shear stress during cooking. There was no significant difference between the flours of MBs and HR whereas SBs showed lower BD. HR and MBs had higher HPV than SBs ($p \leq 0.05$), indicating that the latter has inferior heat stability.

Table 3A.3 Pasting properties of SBs, MBs and HR flours.

Types of flour	PT (°C)	PV (cP)	HPV (cP)	BD (cP)	SB (cP)	FV (cP)	GH (g)
HR	77.89^{a}	3891^{b}	3474^{b}	417^{b}	3014^{c}	6488^{c}	43.45 ± 1.07^{b}
MBs	78.24^{b}	3819^{b}	3447^{b}	372^{b}	2758^{b}	6205^{b}	42.5 ± 1.08^{b}
SBs	78.59^{b}	3219^{a}	3066^{a}	153^{a}	2457^{a}	5523^{a}	30.96 ± 0.84^{a}

Significant differences ($p \leq 0.05$) exist between the mean values with different superscript in a column.

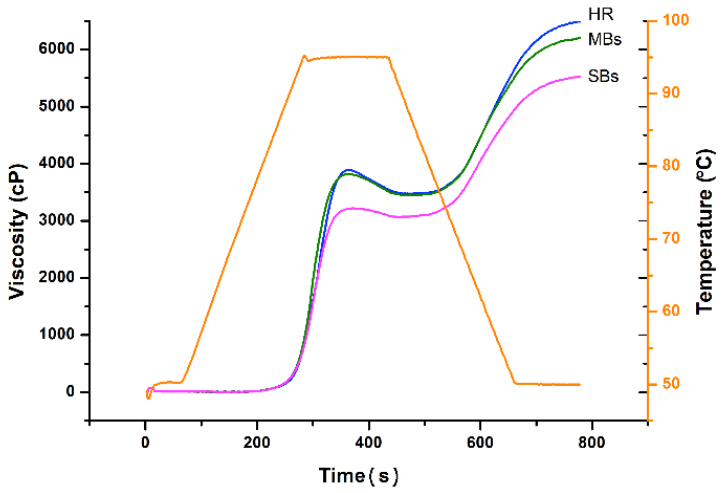

Fig. 3A.2 Pasting properties of SBs, MBs, and HR flours.

FV of a sample reveals its final characteristics, the nature of the starch paste after cooking and cooling in particular. Higher FV indicates harder paste texture. HR showed

highest FV followed by MBs and SBs. SB determines the flour's retrogradation ability that directly correlates with the amylose content i.e., higher the amylose content higher is the SB. Amylose content of MBs was higher than SBs and so was the setback (Table 3.3.), which is totally in agreement with the findings elsewhere [15, 79]. The above findings suggest that SBs flours have lower swelling capabilities (as shown by low PV) and lesser retrogradation capacity than MBs and HR (as shown by low SB). These may be due to many facts reported so far: i) the chemical composition and physical properties of the whole rice grains are different across its length [15, 79]; and ii) amylose and amylopectin ratio in HR compared to brokens [89]. However, it is clear that brokens had lower viscosity when compared to HR, particularly SBs.

The highest RVA gel hardness was found in HR flour (43.45 ± 1.07^{b} g), followed by MBs (42.5 ± 1.08^{b} g) and SBs (30.96 ± 0.84^{a} g) (Table 3A.3). Low gel hardness of SBs can be correlated with high protein and fat content, which have a negative effect on flour gel hardness. From the above results, it is clearly evident that MBs can create good flour paste similar to HR flour paste. Higher gel hardness is a desirable attribute for the rice noodle manufacturing [13, 49].

3A.3.6. Morphological studies

SEM micrographs of grain surface of HR and brokens are presented in Fig 3A.3. SBs (Fig. 3A.3 A2) showed rough surface than MBs (Fig. 3A.3 B2) and HR (Fig. 3A.3 C2) and the reason could be more irregular morphology of the starch particles. Some discrete small particles with irregular shaped (mostly round and oval) were observed which could be the protein bodies [56]. These particles were higher in SBs and MBs than HR. However, starch granules of all three samples were observed to adhere to one another and present in the form of clumps that is indicative of the proteins and lipids binding with starch granules.

Micrographs of the cross-sectional view of HR and brokens were presented in the Fig. 3A.4. It was evident that the SBs fraction (Fig. 3A.4 A1) was smaller than MBs (Fig. 3A.4 B1) and HR (Fig. 3A.4 C1) grain is larger and wider in size. Starch is more closely packed in the protein matrix in all the samples. SBs micrograph reveals smaller starch granules than MBs and HR, implying that the size and shape of endosperm cells may be related to grain size. The micrographs show that the cracks are present in the starchy endosperm of all the three rice kernel sections (Fig. 3A.4. A3, B3 & C3). However, HR (Fig. 3A.4. C2) showed wider radial cracks than MBs and SBs. The grain

likely develops cracks as it matures and begins to lose water [85]. Rice kernels may fracture as a result of abiotic stress caused by drying (Fig 3A.4. C2), breakage during milling, ultimately producing brokens of different shape and size [112]. SBs and MBs showed greater extent of rough cross section surface texture in comparison to HR.

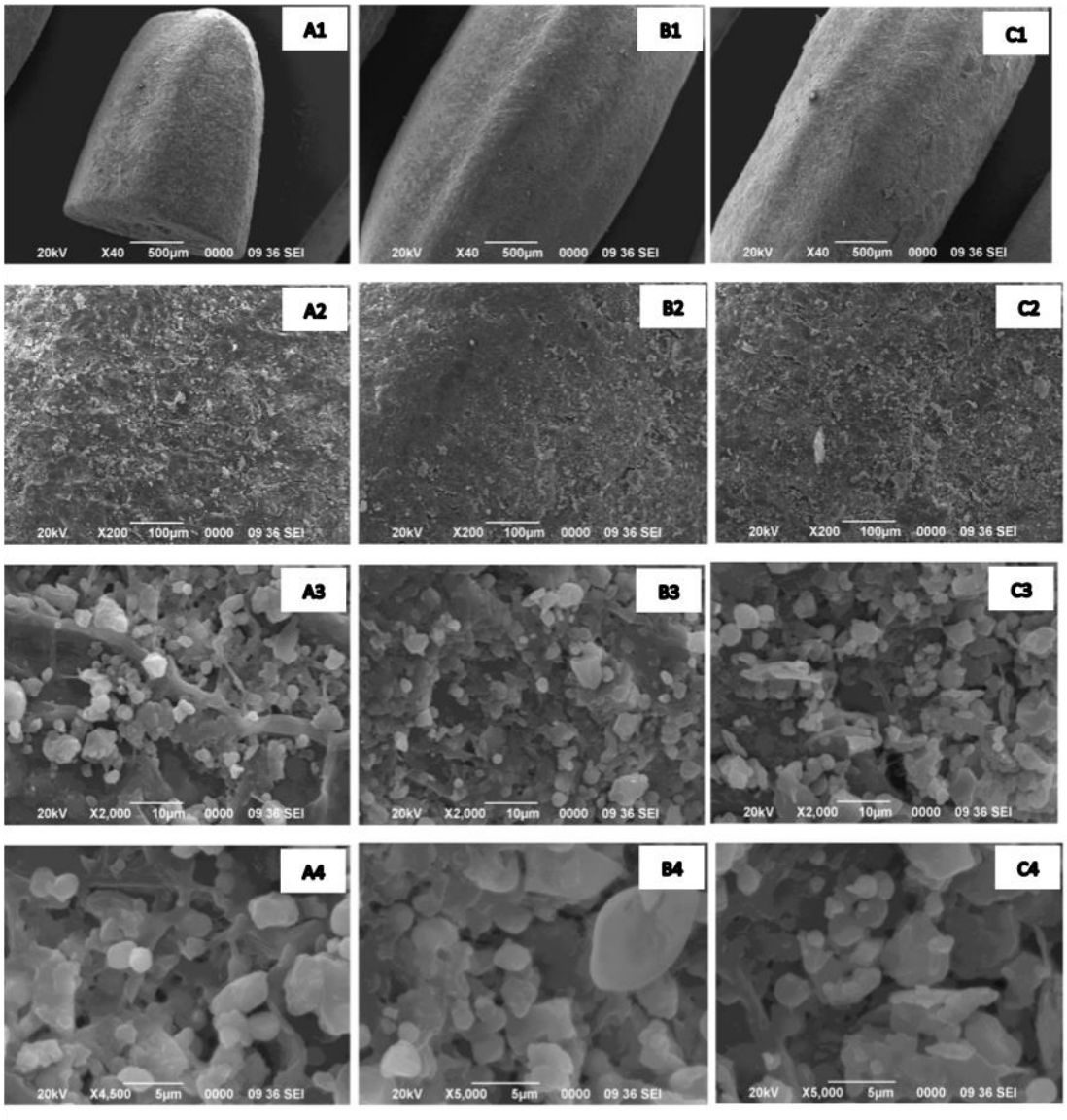

Fig. 3A.3 Scanning electron micrographs of surface of head rice and broken rice. SBs (A1: 40X, A2: 200X, A3: 2000X and A4: 4500X); MBs (B1: 40X, B2: 200X, B3: 2000X and B4: 5000X); and HR (C1: 40X, C2: 200X, C3: 2000X and C4: 5000X).

Micrographs of the flours of HR and brokens are presented in Fig. 3A.5. The rice flours seemed to have rice starch granules with smooth surfaces which are polygonal in shape, as well as a significant number of irregularly shaped particles that were clumped together. Ye et al. [123] reported similar result in rice flour. This is ascribed to the

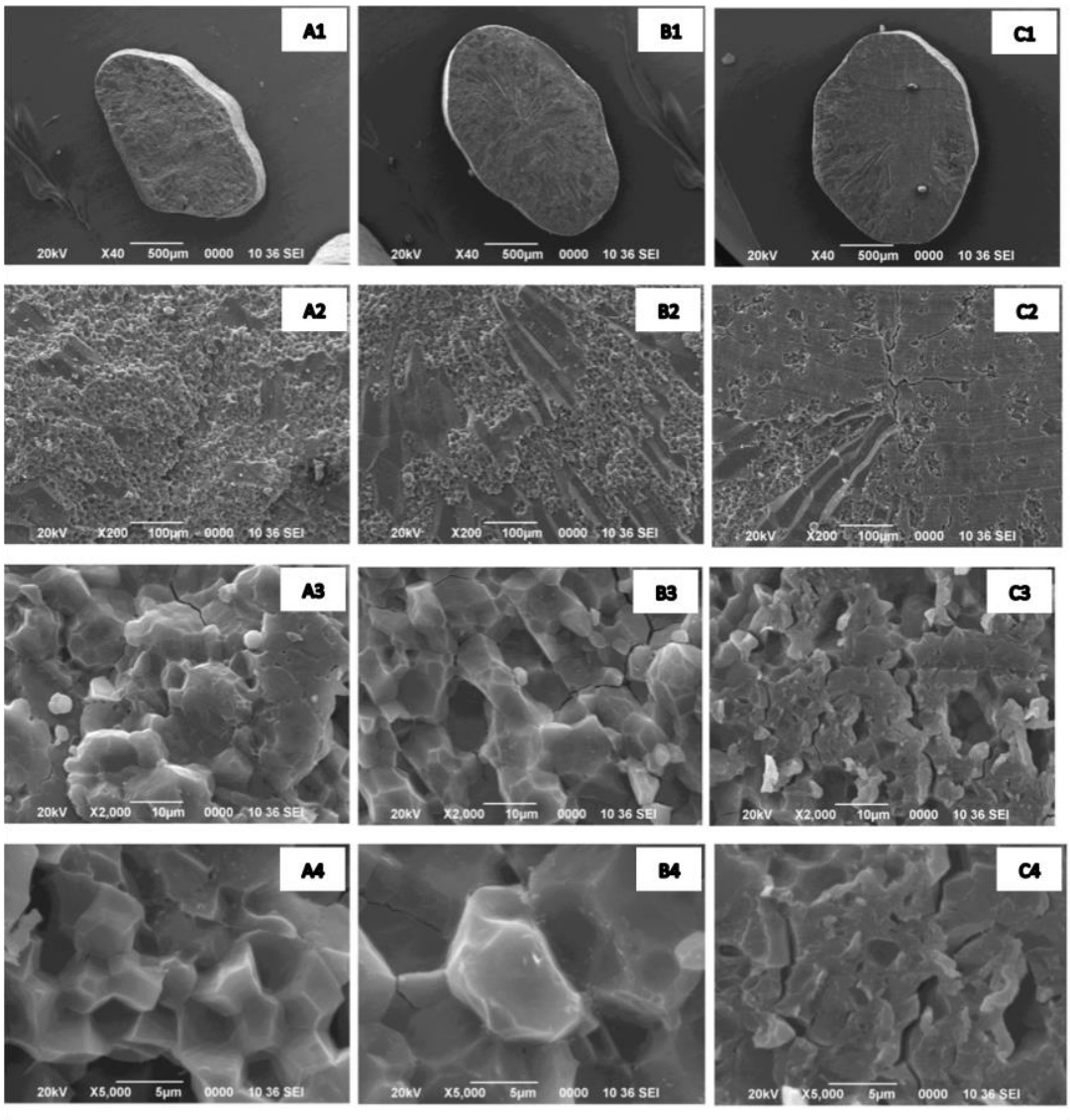

Fig. 3A.4 Scanning electron micrographs of cross section view of head rice and broken rice. SBs (A1: 40X, A2: 200X, A3: 2000X and A4: 5000X); MBs (B1: 40X, B2: 200X, B3: 2000X and B4: 5000X); and HR (C1: 40X, C2: 200X, C3: 2000X and C4: 5000X).

presence of lipids and proteins in the rice flour that are wrapped around the starch granules. Additionally, SBs and MBs contained finer, flat-surfaced particles without the differentiation of starch particles, which indicates the existence of damaged starches. The grinding process causes the starch structure to disintegrate [100]. Higher amount of damaged starch was observed in SBs followed by MBs. HR rice flour is seen to have larger and more uniform starch granules and low level of damaged starch. Due to their high damaged starch content, rice flours with fine starch granules and high amount

of damaged starch are not suitable for the production of high-quality food products like bakery products and noodles, etc. [43, 46].

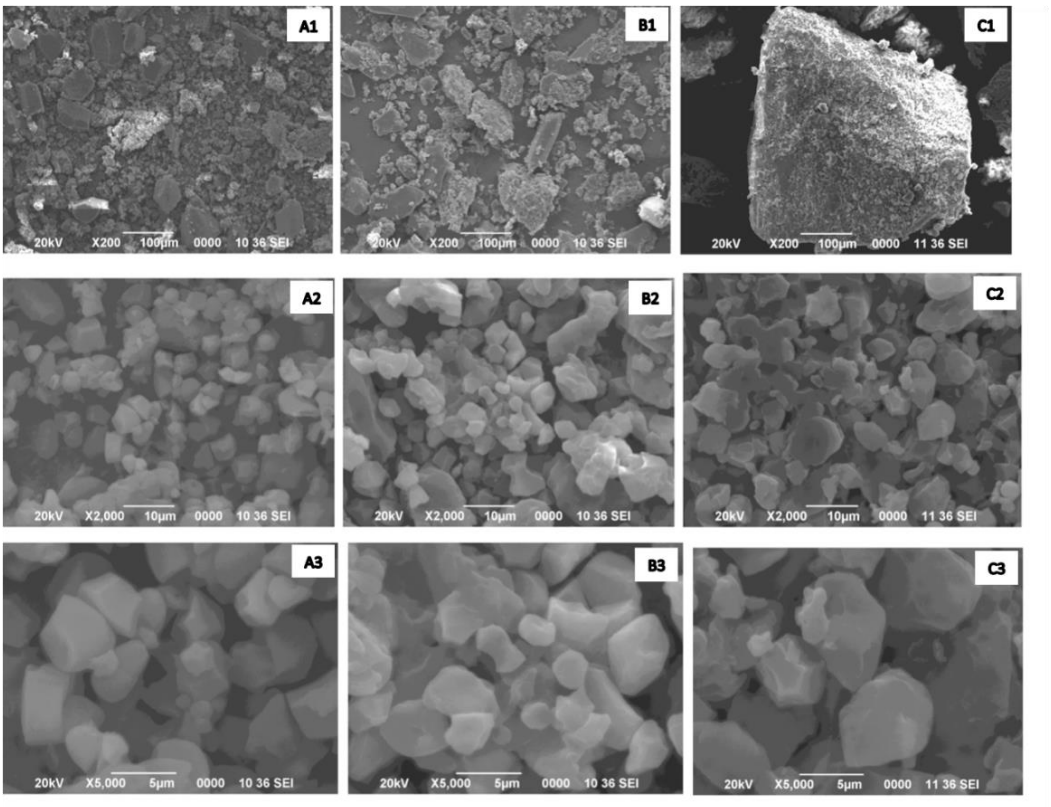

Fig. 3A.5 Scanning electron micrographs of flours of head rice and broken rice. SBs (A1: 200X, A2: 2000X and A3: 5000X); MBs (B1: 200X, B2: 2000X and B3: 5000X); and HR (C1: 200X, C2: 2000X and C3: 5000X).

3A.4. Conclusion

Ranjit rice variety was identified as a grain of medium length and medium shape as per the classification given by Juliano. Size fractionation of the broken rice was conducted using sieve shaker that yielded two main fractions viz. MBs and SBs and the amount of MBs (57.55 ± 4.91%) was higher than SBs (40.49 ± 4.65%). Protein and fat content was significantly higher in SBs than MBs. Starch content was higher in HR than the brokens ($p \leq 0.05$). Among the brokens, MBs had higher starch content than SBs. SBs showed the lowest WAC, SP, SOL and the highest OAC. There were no significant differences in OAC, SP and SOL of MBs and HR. MBs showed better pasting properties than SBs, and similar to HR. The rice flour gel of MBs was harder than the SBs. SBs contain higher damaged starches than MBs and HR as revealed by SEM micrographs. In conclusion MBs showed overall better functional, pasting and starch gel texture and

therefore it was selected for further study and rice noodle development. Thus, size fractionation can be an effective way to identify possible uses of the different fractions of broken rice.

Chapter 3B: The effect of varied particle size of medium broken rice flour on the cooking, textural and sensory qualities of gluten free rice noodles

3B.1. Introduction

Broken kernels are inevitable during rice milling. Broken rice is utilized as animal feed [102] and as a substrate in the brewing process [38] and fermentation [68, 103]. Broken rice flour is also used in processed foods due to its low sodium level, bland taste, ease of digestion, low allergenicity and gluten-free nature [41]. As industrialization and other human activities increase, waste management regulations are becoming more stringent. As a result, there is an urgent need to develop an alternative and efficient method of utilising these by-products to produce high-value products [114]. Moreover, there is an inducement to use the broken rice flour in rice-based food items owing to its low market price. This will encourage the efficient use of locally produced broken rice and also promote its scope of utilization on a larger scale.

Noodles are the most popular and favoured choice among the convenience foods due to their palatable taste, ease in cooking and affordable price. Over the past few years, the noodle sector has consistently expanded, spurring further study on noodles. Noodles are often prepared with wheat flour, but the presence of gluten makes them inappropriate for celiac sufferers [47] and adult women with gynaecological issues such as endometriosis, ovarian cyst etc. As a result, non-gluten grains such as rice corn etc. are the only few options for making gluten free noodles. Rice noodles are usually produced using rice grains having high amylose content, (> 25% amylose) [49], (> 28% amylose) [71]. Noodles can also be prepared using rice variety with intermediate amylose content (< 25%) with comparatively softer texture as compared to noodle from high amylose rice [18]. Rice species with a high amylose content, a low gelatinization temperature, and a firm gel texture are ideal for manufacturing rice noodles [75]. Rice noodles making quality of rice flour is primarily decided by the physicochemical characteristics of starch, which serves as the noodle's structural framework [97]. Cooking, textural, and sensory attributes of rice noodles are the most important features that promote and determine the consumer acceptance. The rice noodle textural mainly depend upon the physicochemical

properties of the rice flour [13, 49] including the flour particle size. Variation in the flour particle size significantly affects the final product texture. Therefore, determining the optimum particle size of the flour is crucial. Yoenyongbuddhagal and Noomhorm, [125] found that rice flour particle size less than 200 mesh was suitable for the production of rice vermicelli with acceptable cooking and textural properties. According to Qian et al. [94] rice flour particle size for the preparation of rice noodles should be in the range of 160-180 μm, since too fine flour can cause transportation issues across the process line, while too large particle sizes can make the products' eating quality coarse and inferior. Kim et al. [61] has reported that smaller particle size (< 100 μm) and bigger particle size (> 250 μm) of rice flours produced higher cooking loss in gluten free noodle slits due to presence of more damaged starch in the former and rough surface in the later. Nagai et al. [81] found good quality noodles when produced using nonglutinous rice flour with 53-212 μm particle size added with 0.5% grain vinegar. Likewise, different authors have suggested suitability of different range of rice flour particle size for rice noodle production. Therefore, along with the physicochemical properties, it is necessary to identify the proper particle size to meet the necessary functionality of the rice flour of a specific rice variety for their intended use.

Limited studies have been made so far on the broken rice of Assam and its application in developing gluten free noodles. It is observed in the previous sub-chapter (Chapter 3A), medium broken rice (1.7-2 mm size) is present in significantly higher quantity and have better physicochemical properties than small brokens (0.85-1.7 mm) next to head rice. Based on the need for developing high value-added product from rice brokens of an intermediate amylose rice of Assam, the objective of this study was to evaluate the suitability of medium broken rice flour of *Ranjit* variety for producing gluten free rice noodles. The impact of particle size of the brokens on functional and pasting properties, and gel hardness of rice flour, and the cooking, textural, and sensory qualities of developed noodles was evaluated.

3B.2. Materials and methods

3B.2.1. Materials

Based on the study discussed in Chapter 3A, the medium brokens of *Ranjit* variety containing 10.00 ± 0.85% moisture, 6.12 ± 0.17% protein, 1.27 ± 0.24% fat, 0.30 ± 0.04% ash, and 24.95 ± 51% amylose content was used for the current investigation.

Medium brokens (MBs) were separated from the whole lot of broken rice using a sieve shaker (Aimil, India) as described in section 3A.2.4. in the previous sub-chapter.

3B.2.2. Preparation of rice flours of different particle size

MBs was washed with tap water, air dried for 1 h and made into flour using a mixer grinder (Philips, model HL7505/00). Rice flour was sieved through a 425 μm sieve and dried at 40 ^{o}C for 24 h in a hot air oven (to reach a safe moisture content for storage) before passing through three laboratory sieves of 212, 180 and 150 μm mesh size in a sieve shaker (Aimil, India), and the three fractions of rice flours obtained were coded as 212F, 180F and 150F, respectively. The flours were then packed separately in airtight containers and stored in the refrigerator until further use.

3B.2.3. Loose bulk density (LBD) and packed bulk density (PBD)

Loose and packed bulk density of rice flour was determined by the method described in section 3A.2.7.1. in the previous sub-Chapter.

3B.2.4. Water absorption capacity (WAC) and oil absorption capacity (OAC)

WAC and OAC of rice flours were determined as per the method given in the section 3A.2.7.2. in Chapter 3A.

3B.2.5. Swelling power (SP) and solubility (SOL)

Swelling power of rice flour and solubility were determined by the detail method explained in the section 3A.2.7.3. in the previous sub-chapter.

3B.2.6. Pasting properties and gel hardness

Rapid Visco Analyzer (RVA Starchmaster 2, Perten Instruments, Hagersten, Sweden) was used to obtain the viscosity profile of rice flours and the RVA gel texture was analysed by texture analyser (TA.XT plus, Stable Micro System Ltd, United Kingdom). The detail methods are same as mentioned in the section 3A.2.8. in the previous sub-chapter.

3B.2.7. Preparation of noodles

Rice noodles were made with rice flour and water adopting slight modification in the method described by [84, 101]. Rice flour was mixed with boiling water and a non-sticky smooth dough was prepared. Dough was extruded using a cylindrical-shaped hand

extruder with a die (0.2 cm diameter pore size). Extruded noodles were collected on a wire mesh and steamed for 30 min. The noodles were cooled to room temperature and air dried for 24 h before being dried in a hot air oven at 40 ^{o}C until moisture content below 12% (wb). Dried noodles were packed in zipper bags and stored in an airtight container at ambient temperature. Market rice noodle (MRN), a proprietary brand was used as the control. Three types of noodles namely 150N, 180N and 212N (where N stands for noodle) were prepared using rice flours, 150F, 180F and 212F, respectively.

3B.2.7.1. Cooking quality

The cooking attributes of noodles were measured as per the method described in American Association of Cereal Chemists (AACC) [1]. Briefly, 2 g of rice noodle strands were cooked in 100 ml boiling water until the middle hard core of noodle strands disappeared (optimum cooking time). Cooked noodles were strained for 5 min. The cooked noodles weight was measured and thereafter dried at 40 ^{o}C overnight. The cooking water was dried in an oven (105 ^{o}C) until it reached a steady weight. The cooking loss, broken rate, rehydration and swelling index of rice noodles were calculated as follows,

$$Cooking\ loss\ (\%) = \frac{Weight\ of\ dried\ residue\ in\ cooking\ water}{Weight\ of\ raw\ noodles} \times 100 \quad \text{Eq. (3B.1)}$$

$$Rehydration\ (\%) = \frac{Weight\ of\ cooked\ noodles - weight\ of\ raw\ noodles}{Weight\ of\ raw\ noodles} \times 100 \quad \text{Eq. (3B.2)}$$

$$Swelling\ index = \frac{Weight\ of\ cooked\ noodles - weight\ of\ cooked\ noodles\ after\ drying}{Weight\ of\ cooked\ noodles\ after\ drying} \quad \text{Eq. (3B.3)}$$

$$Broken\ rate = \frac{Amount\ of\ broken\ cooked\ rice\ noodle\ strands}{Amount\ of\ uncooked\ noodle\ strands} \quad \text{Eq. (3B.4)}$$

3B.2.7.2. Texture measurement

Cooked noodle strands were kept on a strainer and cooled by immersing them in cold water. The cooled noodle strands were placed on a tray, and the texture was measured right away. The texture profile analysis (TPA) of cooked noodles with a length of 25 mm was measured by a texture analyser (TAXT2i, Stable Micro Systems, UK) with 35 mm aluminium cylindrical probe [44]. Each strand of noodles was compressed to 75% deformation at compression speed of 1 mm/s pretest, 5 mm/s test and post-test followed by 5 s holding period before second compression. The textural attributes reported are hardness, adhesiveness, cohesiveness, springiness and chewiness.

For tensile strength, the noodle strands were elongated at a speed of 1 mm/s pre-test speed, 3 mm/s test speed and 10 mm/s post-test speed with a trigger force of 5 g until rupture. The maximum peak force required to resist the breakage of noodle strand was recorded and expressed as tensile strength.

3B.2.7.3. Sensory evaluation

A nine-point Hedonic scale that ranged from 9-like extremely to 1-dislike extremely was utilized for the evaluation of sensory properties [121]. Rice noodles were cooked and served for sensory evaluation to 10 semi-trained panellists, whose age ranged between 20 and 40 years. The panellists were made familiar with the method of sensory evaluation by Hedonic scale and the various textural attributes used for the sensory test. The sensory parameters assessed were colour, appearance, firmness, elasticity, tooth packing, and overall acceptability.

3B.2.8. Statistical analysis

Unless otherwise specified, all analyses were performed in several replicates, with the mean values reported. DMRT was used in the SPSS (28.0.1.1) to determine statistical differences between the means at a significance level of $p < 0.05$.

3B.3. Results and discussion

3B.3.1. Functional properties of rice flours

The results presented in Table 3B.1 reveals that WAC was higher in 150F than 180F and 212F, which can be attributed to the smaller particles size that has larger surface area and therefore, tends to absorb more water [63]. 180F sample had lower OAC and there was insignificant difference between the OAC of 150F and 212F. Since starch lacks non-polar sites like those present in proteins, the OAC is mostly caused by the physical trapping of oil inside the starch molecule [33]. Insignificant difference was found in the loose bulk density of 150F and 180F whereas 212F showed the lowest value. Packed bulk density of 150F was statistically similar to 180F whereas different from 212F. It was evident from the results that the 150F had the highest density.

Swelling power of 150F was significantly higher than 180F and 212F (Fig. 3B.1). Higher swelling power was also reported in 150-180 μm particle size flour than 132-150 μm particle size flour elsewhere [28]. Swelling power refers to hydration capacity of starch under specific conditions. It mostly depends on the structure and amylose/amylopectin ratio as well as the presence of non-carbohydrate components that

impede swelling, such as proteins and lipids.

Table 3B.1 Water and oil absorption capacity, loose and packed bulk density of medium broken rice flours with different particle size.

Rice flours	Water absorption capacity (g/ml)	Oil absorption capacity (g/ml)	Loose bulk density (g/ml)	Packed bulk density (g/ml)
150F	2.35 ± 0.003^{c}	2.23 ± 0.01^{b}	0.69 ± 0.02^{b}	0.96 ± 0.01^{b}
180F	2.33 ± 0.002^{b}	2.15 ± 0.03^{a}	0.68 ± 0.01^{b}	0.94 ± 0.02^{ab}
212F	2.32 ± 0.05^{a}	2.21 ± 0.02^{b}	0.63 ± 0.01^{a}	0.92 ± 0.01^{a}

Results are represented as mean ± SD (n = 3); significant differences ($p \leq 0.05$) exist between the mean values with different superscript in a column.

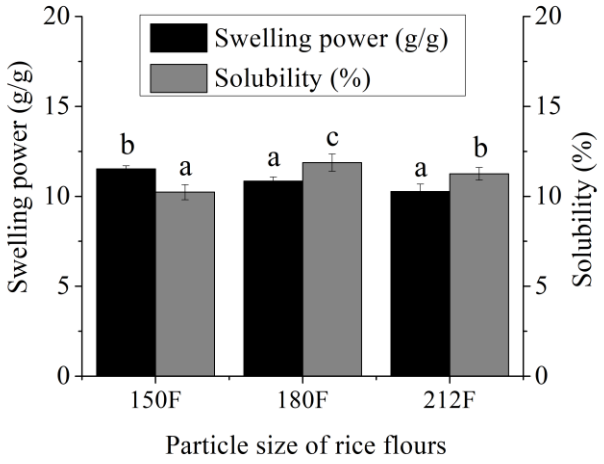

Fig. 3B.1 Swelling power and solubility of medium broken rice flours with different particle size. Values presented in the bar diagram are the means of triplicate determinations. The error bars are the standard deviation of mean values. Significant difference ($p \leq 0.05$) exists between the means in a column group with different letters (a-c) on the column.

Solubility accounts for the amount of dispersed soluble amylose after heating in an aqueous solution. Solubility of 150F, 180F and 212F was significantly different, and 180F recorded the maximum solubility (150F < 212F < 180F). Thus, flour particle sizes do not seem to influence the distribution and release of soluble solids, according to the obtained results. Similarly, no relationship was found between flour of particle size less than 250 μm and solubility index by several researchers [4, 28]. On the contrary, an increase in solubility with reduction in particle size of flour was also reported [35].

3B.3.2. Pasting properties and gel hardness

The pasting properties of rice flours are presented in Table 3B.2. and Fig. 3B.2. Pasting temperatures (PT) of 150F, 180F and 212F was 70.16 ± 2.06 ^{o}C, 77.01 ± 1.32 ^{o}C and 78.34 ± 1.07 ^{o}C, respectively. The low PT of 150F may be ascribed to the ability of smaller particle size flour to rapidly absorb water and enable its starch granules to swell faster, allowing for easier gelatinization of starch at lower temperatures [84]. PV, HPV, SB and FV of 150F was significantly higher than 180F and 212F. PV denotes the maximum viscosity attained by starch after gelatinization under specified conditions [34]. The high PV of 150F paste indicates that starch granules have a high tendency to swell readily owing to the large surface area of small particle size rice flour [62] before their physical disintegration because of greater breakdown due to lower heat and shear stress tolerance during heating [50]. The higher PV of 150F could make it a better alternative for gluten-free food formulations [3].

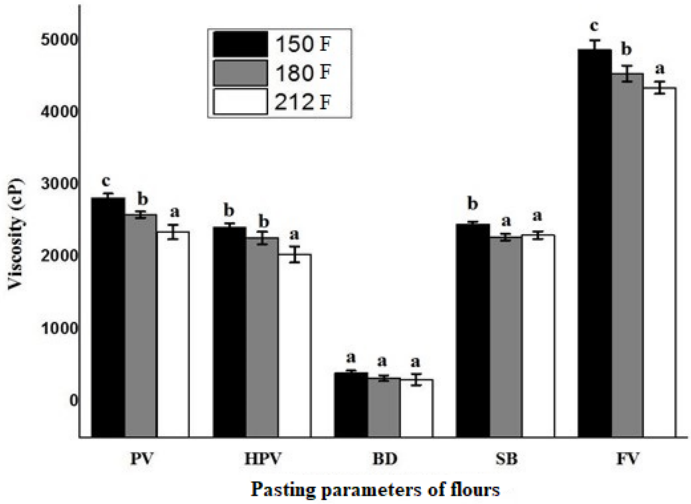

Fig. 3B.2 Pasting properties of medium broken rice flours with different particle size: PV: Peak viscosity; HPV: hot paste viscosity; BD: breakdown; SB: setback; and FV: final viscosity. Values presented in the bar diagram are the means of duplicate determinations. The error bars are the standard deviation of mean values. Significant difference ($p \leq 0.05$) exists between the means in a column group with different letters (a-c) on the column.

Table 3B.2 Pasting properties and gel hardness of medium broken rice flours with different particle size.

Rice flours	Pasting temperature (ºC)	Peak viscosity (cP)	Hot paste viscosity (cP)	Breakdown (cP)	Setback (cP)	Final viscosity (cP)	Gel hardness (g)
150F	70.16±2.06^{a}	2825.37±61.34^{c}	2422.71±52.71^{b}	402.66±29.47^{a}	2462.67±35.73^{b}	4885.38±128.37^{c}	42.64±0.47^{c}
180F	77.01±1.32^{b}	2597.52±44.57^{b}	2270.26±86.26^{a}	327.26±41.93^{a}	2282.56±44.81^{a}	4552.82±110.82^{b}	41.62±0.24^{b}
212F	78.34±1.07^{b}	2355.25±100.35^{a}	2044.18±111.63^{a}	311.07±78.46^{a}	2312.55±51.67^{a}	4356.73±84.14^{a}	39.73±0.45^{a}

Results are represented as mean ± SD (n = 2); significant differences ($p \leq 0.05$) exist between the mean values with different superscript in a column.

HPV is the measure of stability of hot paste, which is affected by granule swelling, the amylose release rate and the formation of amylose-lipid complex [104]. High HPV of 150F indicates better heat stability as compared to flours of 180F and 212F. In general, a high HPV indicates reduced cooking loss and good eating quality [13]. Higher SB value in 150F revealed that the flour will form firm paste/gel. Rice flour exhibiting high setback is appropriate for stick rice noodles and extruded products [3]. FV is mostly governed by the retrogradation of the soluble amylose, fragments of swollen granules during cooling and a high FV relates to a strong resistance to shear. There was no statistical difference in the BD of the three flours. These pasting profile of the three samples revealed that rice flour with 150 μm gelatinized earlier and displayed more firm gel forming ability than flour samples with larger particle sizes [84].

Significant difference in GH was observed where the 150F had the highest GH (42.64 ± 0.47 g) followed by 180F (41.62 ± 0.24 g) and 212F (39.73 ± 0.45 g) (Table 3B.2). An inverse relationship between the gel hardness and flour particle size has been reported [84]. The amount of amylose released into the starch gel increased when the flour particle size was smaller, resulting in quick retrogradation and consequent increase in gel hardness. The dominant factor, which well correlates with the actual texture of cooked noodles, is the gel hardness of the flour, which could be used as a preliminary indicator for predicting the rice noodle quality [13, 124].

3B.3.3. Cooking properties of rice noodles

The results of cooking properties presented in Table 3B.3 showed that the prepared noodles had significantly higher cooking time than the MRN with no significant difference among the them. There was significant difference in cooking loss between the marketed and test noodle samples; also, among the test noodle samples as well. Cooking loss is a significant factor in noodles, since it measures the amount of irrecoverable particulates in the cooking water. Cooking loss below 10% is regular and acceptable whereas above 10% is considered poor and unfavourable because it will cause turbidity in boiling water and a sticky mouth feel with poorer cooking tolerance [13, 44, 70].

The three types of noodles resulted in relatively higher cooking loss than MRN. However, 150N showed significantly lower loss than 180N and 212N noodles. It was also observed that the noodle strands, which were initially sticked together during

Table 3B.3 Cooking properties of rice noodles prepared using medium broken rice flours with three different particle size.

Noodle Samples	Cooking time (min)	Cooking loss (%)	Rehydration (%)	Swelling index	Broken rate (%)
MRN	5.5 ± 0.1^{a}	5.05 ± 0.18^{a}	167.83 ± 6.32^{a}	1.34 ± 0.05^{a}	0^{a}
150N	6.3 ± 0.4^{b}	9.57 ± 1.12^{b}	198.89 ± 5.13^{b}	1.88 ± 0.06^{b}	8.33 ± 1.20^{b}
180N	6.5 ± 0.2^{b}	12.03 ± 1.22^{c}	202.26 ± 8.82^{b}	1.96 ± 0.05^{b}	12.09 ± 2.71^{c}
212N	6.8 ± 0.5^{b}	16.42 ± 1.05^{d}	210.67 ± 6.04^{c}	2.21 ± 0.08^{b}	15.09 ± 1.48^{d}

Results are represented as mean ± SD (n = 3), Significant differences ($p \leq 0.05$) exist between the mean values with different superscript in a column. MRN: marketed rice noodle, 150N: Noodle made from 150F, 180N: noodle made from 180F and 212N: noodle made from 212F.

drying, remained attached during cooking. Strands hardly get separated and some strands were broken and solids were also released while trying to separate the attached strands during cooking. This may also have increased the cooking loss. Higher cooking loss (9-19%) of rice noodles were reported elsewhere [34]. Cooking loss increased with the increasing particle size of the flour which was also reported by some authors [13, 45]. Moreover, cooked strands of 180N and 212N showed rougher surface, which might have interacted with water in higher extent during cooking resulting in higher cooking loss [61]. High cooking loss was reported in noodles made from flour with particle size less than 100 μm and also with flour having particle size above 250 μm [61]. This suggested that the particle size of rice noodles should be in between the 100 to 250 μm for better quality noodle. Here, 150N noodle was acceptable due to significantly lower cooking loss (9.57%) than its other two counterparts.

Cooking attributes of noodles are associated with rehydration and the swelling index [57] that also affects the texture of noodles. Rehydration is the weight ratio of cooked noodles to uncooked noodles. Inadequate rehydration normally results in a hard and gritty texture, whereas excessive water intake frequently results in a mushy and sticky noodle [49]. Proper rehydration in noodles is therefore necessary. In the present work, all three noodles outperformed MRN in terms of rehydration in the following order: 150N < 180N < 212N. 150N noodle was firmer and 212N noodle was softer. A similar pattern of change was found in the swelling index, which increased as the particle size of rice flour increased. Contradictory results of low water uptake associated with high solid loss in cooked noodles was reported [45]. Cooked MRN had no broken rate, however prepared noodles had a significantly different broken rate where 150N showed the lowest and 212N showed the highest broken rate. This might be explained by the variations in the hardness and strength of flour paste of different particle size rice flours. Thus, 150N showed relatively better cooking properties among the three noodles. However, the cooking properties of the three noodle samples did not compare well with MRN. The findings suggested the need to improve the characteristics of the rice flours to reach similar cooking properties as the MRN.

3B.3.4. Textural properties

The texture of rice noodles was significantly influenced by the particle size of flour (Table 3B.4.). Hardness was significantly affected by the particle size; the highest and the lowest hardness were exhibited by 150N and 212N, respectively. This is supported by the results obtained for wheat flour noodle [72]. The adhesiveness, springiness, and chewiness of the three rice noodles did not differ statistically, proving that particle size had no influence on these qualities. Increase in chewiness of wheat noodle with decrease in the particle size of the flour was reported [46, 72]. The cohesiveness of 150N was statistically similar to 180N but different from 212N. There was no significant difference between the tensile strength of 150N and 180N whereas 150N had significantly higher tensile strength than 212N. Increased proportions of smaller granules are responsible for increasing the elastic behaviour [51]. All the test noodles samples showed substantially low breaking distance than the MRN ($p < 0.05$). MRN exhibited far higher values for textural attributes than the prepared noodles ($p < 0.05$).

Thus, all the three noodles were inferior to MRN in the textural properties due to the difference in processing conditions and variety of rice used. However, a pattern of increased textural attributes on reduction of particle size of flours was observed. The reason for this may be due to more uniform and stronger gelatinization and proper retrogradation process in noodles with smaller particle size [125]. This is also consistent with the results of RVA pasting properties that exhibited lower pasting temperature and higher paste viscosities by 150F. Gelatinized starches of rice flour acts as binding agent that is necessary to achieve required structure in the rice noodle to improve the cooking as well as the textural properties to an optimum level [69, 84, 125]. As a result, the flour samples with 150F produced rice noodle with better textural quality than the 180F and 212F noodles.

3B.3.5. Sensory quality

Sensory analysis results of the rice noodles presented in Fig. 3B.3 shows no significant difference in the colour of the test noodles whereas MRN showed significantly more whiteness and translucency (visualization by naked eyes). This indicated that the panellists preferred rice noodles which are white and translucent. 150N scored significantly higher score in appearance and firmness than 180N and

Table 3B.4 Textural properties of rice noodles prepared from medium broken rice flours with different particle size.

Noodle Samples	Hardness (g)	Adhesiveness (g.s)	Springiness (mm)	Cohesiveness	Chewiness (g.mm)	Tensile (g)	Breaking distance (mm)
MRN	5427.79±59.17^{d}	-17.23±1.72^{b}	0.966±0.01^{a}	0.825±0.01^{c}	4324.55±81.62^{b}	54.73±0.61^{c}	8.67±0.17^{d}
150N	3920.53±45.12^{c}	-1.99±1.32^{a}	0.82±0.01^{a}	0.69±0.02^{b}	2220.14±13.32^{a}	33.2±1.00^{b}	5.45±0.24^{a}
180N	3673.61±76.11^{b}	-1.66±1.27^{a}	0.77±0.02^{a}	0.68±0.04^{ab}	1948.93±111.02^{a}	32.12±1.00^{ba}	5.3±0.32^{a}
212N	3533.84±10.05^{a}	-1.69±0.41^{a}	0.82±0.03^{a}	0.64±0.004^{a}	1850.91±85.83^{a}	31.33±0.57^{a}	5.14±0.19^{a}

Results are represented as mean ± SD (n = 5), Significant differences ($p \leq 0.05$) exist between the mean values with different superscript in a column. MRN: marketed rice noodle, 150N: Noodle made from 150F, 180N: noodle made from 180F and 212N: noodle made from 212F.

212N. Noodles that are too firm or too soft when cooked are undesirable [2, 121]. Flour with higher amylose content gives more firmness. 150N scored comparatively higher elasticity than 180N and 212N ($p > 0.05$). Tooth packing refers to the amount of noodle that remain on the teeth after chewing one strand of noodle. [121]. MRN scored the highest on tooth packing indicating less amount of chewed noodle strand left on the teeth followed by 150N, 180N and 212N. The sensory analysis results indicate that the overall acceptability of the noodles was comparable, ranging from like slightly to like moderately (6.2 to 7). 150N scored significantly higher than 180N and 212N in overall acceptability. Rice noodle made from 150F received sensory scores above 6 for all the sensory attributes, thus indicating that the panellists liked 150N among the three rice noodles.

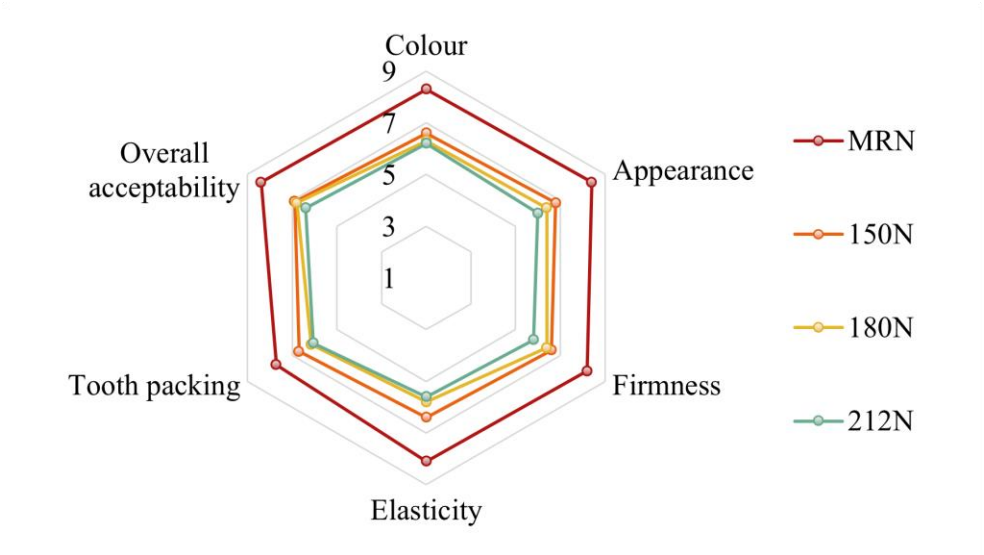

Fig. 3B.3 Sensory evaluation of rice noodles made from medium broken rice flours of different particle size. MRN: marketed rice noodle, 150N: Noodle made from 150F, 180N: noodle made from 180F and 212N: noodle made from 212F.

3B.4. Conclusion

In the present study, water absorption capacity, oil absorption capacity, swelling power, solubility, pasting properties, and gel hardness of the rice flour were significantly influenced by the particle size of the flour. Flour with the 150 μm particle size showed better pasting quality for noodle processing. Noodles made from 150 μm size flour had considerably less cooking loss, higher textural attributes and higher sensory scores as compared to the noodles made from larger particle size flours. Accordingly, rice noodles made from 150 μm indicated acceptable cooking,

textural, and sensory properties than the other two noodles. This study established the rationale for the efficient use of medium broken rice flour of Assam variety in the manufacture of gluten free rice noodles. However, the cooking loss and broken rates of the noodles made from 150 μm size flour were also marginally low than the maximum limit of a good quality rice noodle. Thus, the obtained results indicated the scope for improvement in the noodle making qualities of the flour or starch isolated from it.

Chapter 3C: Process optimization of osmotic pressure treatment and heat moisture treatment for rice starch isolated from medium broken rice

3C.1. Introduction

As observed in Chapter 3B, the prepared noodles had significantly high cooking loss, noodle strand breakage, low sensory scores and textural attributes than the market noodle. Therefore, the need for some improvement in the eating quality of the noodle arises to compete with the commercial rice noodle. Medium broken rice possesses significant amount of starch as observed in Chapter 3A (Table 3A.1). Thereby, it would be a cheaper source of starch as compared to whole grain because the price of broken rice is far less than head rice. The quality of rice noodles is primarily determined by the physicochemical qualities of rice starch, which contribute to its unique structural network [118]. Therefore, improving the functionality of the isolated starch can be an effective way to enrich the overall quality of the final noodle.

Rice starch is a crucial element in rice noodle manufacture, and its functions may be significantly enhanced by using appropriate modification methods [10]. HMT and OPT are two physical modification approaches that showed similarity in the changes in the modified starches as seen in potato, sago and corn starch [9, 90-92]. During HMT, starch is adjusted to a low moisture content of 10-30% and heated at high temperatures (80-120 ^{o}C) for a specific period of time (15 min- 16 h) [5]. OPT employs an excess of saturated sodium sulphate solution that raises the osmotic pressure of the solution [90-92], thereby decrease the availability of free water owing to the formation of hydration shell by sulphate ions with water [52]. Thus, water penetration into the granule is inhibited, resulting in retardation of gelatinization of starch molecules during the treatment.

Response surface methodology (RSM) is an efficient statistical approach. It is based on fitting a polynomial equation to experimental data to characterize the behaviours of a data set with the goal of generating statistical predictions [12]. RSM involves a smaller number of experimental runs. Using different starch sources, including rice starch, RSM has been used to optimize steam-heat moisture treatment [31], dry-heat moisture treatment [30], and annealing [18]. However, this is the first time to study the optimization of OPT treatment conditions on rice starch. Rice noodle qualities have been reported to correlate with RVA paste viscosities, gel texture, swelling power, and solubility of the starch in rice flour [13, 49], and are therefore key functionalities for rice noodle production. HMT and OPT can reduce granule swelling, delay gelatinization, raise the stability of starch paste, and increase the hardness of gel [65]. HMT starch has been used to improve rice noodle quality in some previous studies [18, 49]. However, investigation on the usability of OPT starch in rice noodle development is scanty. Therefore, it was hypothesized that OPT could bring similar changes in the rice starch as HMT and consequently be applied in the improvement of overall quality of rice noodle by partially replacing the rice flour with modified starch.

Based on the aforementioned background, the goal of this research was to find the best treatment conditions for OPT and HMT for rice starch in order to predict the enhancement in their physicochemical properties. The OPT and HMT process conditions, namely temperature, time, and moisture content, were optimized in the current study using RSM by face-centered central composite design (FCCD).

3C.2. Materials and methods

3C.2.1. Starch extraction

Rice starch was extracted from medium brokens (MBs) of *Ranjit* variety using an alkaline protein extraction technique as reported previously by Sodhi and Singh [106] with slight modification. To soften the broken endosperm, the broken rice grains (1 kg) were steeped in 1 L 0.2% NaOH solution for 18 h at 4 ^{o}C. After draining the steeped liquid, a mixer grinder (Philips HL1606 500-Watt) was used to turn the broken endosperm into slurry. The slurry was dispersed in 1L of 0.2% NaOH once again, mixed for 10 min and allowed to rest for 6 h. The turbid supernatant was drained off and the slurry was centrifuged for 5 min at 1200 × g. The alkaline washing procedure was repeated until the supernatant passed the Biuret test. Sediment was

mixed with distilled water and passed through 425, 300, and 150 μm sieves before being rinsed with water several times until the phenolphthalein-colored supernatant was no longer pink. After centrifugation, the starch sediment was dried at 40 ^{o}C for 24 h. Dried starch was powdered and sieved through 150 μm sieve, kept in airtight container and stored at 8 ^{o}C until it was needed. All the reagents were analytical grade and obtained from Merck Co. Ltd.

3C.2.2. Proximate analysis

Standard AOAC techniques of analysis were used to assess the proximate components (moisture, protein, fat, and ash) of the rice starch [7]. The detail methods are given in the Chapter 3A at section 3A.2.6.1. for moisture content, 3A.2.6.2. for protein content, 3A.2.6.3. for fat content and 3A.2.6.4. for ash content.

The sodium content of native rice starch (NRS) and modified rice starches was measured using an atomic absorption spectrometer (AAS ICE-3500, Thermo Scientific, UK) [8]. 0.5g starch sample was taken in digestion tube to which 10 ml of solution mixture of concentrated nitric acid and concentrated sulphuric acid in the ratio of 1:3 was added. The mixture was heated at 350 ^{o}C until the liquid mixture turns clear and light yellowish in colour. The digested liquid so obtained was used for the determination of sodium content in atomic absorption spectrometer.

3C.2.3. Experimental design for optimization

FCCD was used to generate the experimental designs for OPT and HMT treatments of the treatment temperature and treatment time at three levels for OPT, and of treatment temperature, moisture content of starch, and treatment time at three levels for HMT as listed in Table 3C.1 were used. FCCD was used to establish the association between variables such as final viscosity (FV), setback viscosity (SB), gel hardness (GH) of rice starch gel, swelling power (SP), and rice starch solubility (SOL). The goal of the optimization was to obtain higher FV, SB, GH, and lower SP and SOL. According to Eq. (3C.1), OPT had four factorial points, four axial points, and five centre points, resulting in 13 sets of experiments. HMT had eight factorial points, six axial points, and six centre points, resulting in 20 sets of experiments.

$$N = n_f + n_a + n_c \qquad \text{Eq. (3C.1)}$$

where, N = total number of experiments, n_f = number of factorial design experiments carried out at +1 and -1 as the coded variables of independent variables ($n_f = 2^{\text{no. of independent variables}}$), n_a= number of experiments carried out at $+a_m$ and $-a_m$ (n_a = 2×no. of independent variables), and n_c = number of experiments carried out at the centre point where the values of coded variables is zero. The experiments were conducted in a random order to reduce the effects of unforeseen variability in response due to external factors.

A second-order polynomial function, Eq. (3C.2), was utilized to fit the response taken into consideration from experimental designs in order to determine the optimum process conditions [60]. Where, Y was the predicted response variable, and β_o, β_i, β_{ii}, β_{ij} were the regression coefficients of variables for intercept, linear, quadratic and interaction terms, respectively. X_i and X_j were the codes of independent variables.

$$Y = \beta_0 + \sum_{i=1}^{3} \beta_i X_i + \sum_{i=1}^{3} \beta_{ii} X_i^{\,2} + \sum_{i=1}^{2} \sum_{j=i+1}^{3} \beta_{ij} X_i X_j + e \qquad \text{Eq. (3C.2)}$$

Table 3C.1 Experimental ranges and levels of coded and actual values of independent variables of FCCD for OPT and HMT of rice starch.

Independent Variables	**Symbols**	**Factor levels**		
		-1	0	1
OPT				
Temperature (°C)	X_1	100	110	120
Time (min)	X_2	15	30	45
HMT				
Temperature (°C)	X_1	100	110	120
Time (min)	X_2	15	30	45
Moisture content (%)	X_3	20	25	30

By using correlation coefficients of determination (R2) and coefficient of variation (CV), the interaction between the independent variables was examined. For the model fitting, both graphical and numerical studies were carried out, and an analysis of variance (ANOVA) was used to determine the statistical significance of

the regression coefficients. Using Design Expert 6.0.8 software, a multiple response optimization method was used to calculate the optimal solution.

3C.2.3.1. Preparation of OPT starch

OPT starch was prepared from rice starch using saturated sodium sulphate solution as described in [90-92] with slight modification. Briefly, 100 g of sample was mixed with 200 ml of saturated sodium sulphate solution. Rice starch and salt solution mixture was heated as per the experimental runs of the design by applying calculated osmotic pressure of 327.5 bar (100 °C), 336.25 bar (110 °C) and 345 bar (120 °C). Starch samples were washed with distilled water several times and centrifuged after each wash to remove the residual salt. Presence of salt residue was tested by 0.1 M Barium Chloride solution. Starch sediment was dried at 40 °C for 24 h, ground using mortar and pestle, sieved through 150 μm sieve, packed in an airtight glass container and kept in a refrigerator until further analysis.

3C.2.3.2. Preparation of HMT starch

HMT starch was prepared from rice starch using the method as described in [90-92] with slight modification. Rice starch moisture content was adjusted within the range of 20–30% (wb) by adding calculated amount of distilled water. The moistened starch was equilibrated overnight before being heated in a hot air oven in accordance with the FCCD design (Table 3C.2.). Finally, the starch sediment was dried at 40 ^{o}C for 24 h, powdered with a mortar and pestle, and sieved through 150 μm sieve, packed in an airtight glass container and kept in a refrigerator until further analysis.

3C.2.3.3. Pasting properties

The pasting characteristics of modified rice starch as per the method described in Chapter 3A, section 3A.2.8.

3C.2.3.4. Gel hardness

After the RVA test, the gelatinized starch water slurry in the canister was tested for the gel hardness as per the method explained in the section 3A.2.8. of Chapter 3A.

3C.2.3.5. Swelling power and solubility

Swelling power and solubility of starch were evaluated by following the method explained in the section 3A.2.7.3. (Chapter 3A).

3C.3. Results and discussion

3C.3.1. Proximate composition and sodium content

The native rice starch used in the current study contained 10 ± 0.05 (%, wb) moisture, 0.048 ± 0.002 (%, db) protein, 0.01 ± 0.001 (%, db) fat, and 0.17 ± 0.05 (%, db) ash content. Native rice starch had a sodium content of 2.6 mg/100 g, while OPT and HMT starches had sodium content of 2.7 mg/100 g and 2.6 mg/100 g, respectively, under optimum process conditions. These findings suggest that there was no significant change in sodium content after OPT, which can be attributed to the lack of any interaction between the starch and sodium sulphate solution that was used for OPT treatment.

3C.3.2. Optimization of OPT and HMT processes

3C.3.2.1. Fitting models

All OPT and HMT experiments were carried out within the ranges and levels of the independent variables specified in Table 3C.1. The complete experimental results and the predicted values of the responses for OPT and HMT are presented in Table 3C.2 and Table 3C.3, respectively. The experimental results were fitted to a second-order polynomial model to demonstrate the correlation between the independent factors and responses that is given in Eq. (3C.2). ANOVA was used to determine the significance of each model term, and the findings for OPT and HMT are shown in Table 3C.5 (final viscosity), Table 3C.6 (setback viscosity), Table 3C.7 (gel hardness), Table 3C.8 (swelling power), and Table 3C.9 (solubility). The response variables are statistically significant if the model terms have a high F value and a low p-value. At 95% confidence level, F value and p-value were monitored, and all five responses in both processes demonstrated significance of the proposed model, as evidenced by a low p-value of 0.05. The R^2 and significance of lack-of-fit were employed to assess the model's fitness. The findings validated the model's fitting with $R^2 > 0.85$, indicating that at least 85% of the predicted values matched the actual values. The non-significant lack-of-fit ($p > 0.05$) for all the responses for OPT and HMT signifies that the designed model were adequate to predict the response

Table 3C.2 Experimental design matrix of OPT of rice starch with experimental and predicted responses.

Run Order	Coded levels of independent variables		Experimental value of responses					Predicted value of responses				
	X_1	X_2	Y_{FV}	Y_{SB}	Y_{GH}	Y_{SP}	Y_{SOL}	Y_{FV}	Y_{SB}	Y_{GH}	Y_{SP}	Y_{SOL}
1	120	30	4824.21	2449.78	72.49	18.04	12.23	4796.94	2539.04	72.56	17.94	12.17
2	110	45	4600.00	1664.78	73.93	13.64	11.84	4531.02	1754.04	73.22	13.89	11.78
3	110	30	4573.65	2142.00	69.16	13.71	9.6	4522.57	2001.10	70.16	13.50	10.19
4	120	45	4841.81	2432.36	74.9	19.26	13.03	4879.89	2387.73	75.47	19.17	13.06
5	110	30	4527.52	2066.00	70.32	13.77	10.6	4522.57	2001.10	70.16	13.50	10.19
6	120	15	4622.98	2255.86	67.47	17.46	15.74	4612.18	2211.23	66.83	17.64	15.77
7	110	30	4467.00	1937.00	69.56	13.98	10.7	4522.57	2001.10	70.16	13.50	10.19
8	100	45	4454.31	1752.86	71.77	15.19	10.53	4485.22	1708.23	71.92	15.03	10.56
9	110	15	4383.54	1679.78	62.58	14.33	13.15	4412.31	1769.04	64.27	14.04	13.09
10	110	30	4528.48	2084.00	72.7	13.16	10	4522.57	2001.10	70.16	13.50	10.19
11	100	30	4564.21	1961.78	67.79	15.39	8.33	4551.27	2051.04	68.70	15.46	8.27
12	100	15	4533.48	1959.36	63.72	16.74	10.44	4515.51	1914.73	62.66	16.84	10.47
13	110	30	4476.00	1955.00	70.04	12.82	9.9	4522.57	2001.10	70.16	13.50	10.19

X_1 - Temperature, X_2 - Time; Y_{FV} - Final viscosity, Y_{SB} - Setback viscosity, Y_{GH} - Gel hardness, Y_{SP} - Swelling power, and Y_{SOL} - Solubility

Table 3C.3 Experimental design matrix of HMT of rice starch with experimental and predicted responses.

Run Order	Coded levels of independent variables			Experimental value of responses					Predicted value of responses				
	X_1	X_2	X_3	Y_{FV}	Y_{SB}	Y_{GH}	Y_{SP}	Y_{SOL}	Y_{FV}	Y_{SB}	Y_{GH}	Y_{SP}	Y_{SOL}
1	120	15	20	4926	3114	74.80	15.12	8.8	4805.14	2946.90	74.61	14.70	8.37
2	120	45	30	5893	3667	69.27	13.22	4.5	5888.64	3580.40	69.18	12.88	4.41
3	110	30	30	5137	3233	72.08	10.34	7	5131.73	3224.49	72.93	10.73	6.76
4	110	30	20	4797	2920	79.54	11.06	8.2	4898.73	2962.09	78.72	11.97	9.22
5	110	15	25	3876	2046	77.20	11.35	7.3	4103.73	2198.49	78.26	11.91	7.74
6	120	30	25	5027	3083	71.36	13.13	7.1	5424.93	3465.89	71.35	13.68	6.88
7	110	30	25	5065	3124	75.02	12.15	7.9	4878.18	2976.47	76.65	11.42	7.66
8	110	30	25	4726	2819	75.50	11.14	8.1	4878.18	2976.47	76.65	11.42	7.66
9	120	15	30	5157	3228	70.24	13.96	5	4989.14	3135.80	69.91	14.15	5.51
10	100	30	25	5123	3145	74.88	13.50	7.5	4821.53	2795.69	74.91	14.25	8.50
11	110	45	25	5166	3258	79.94	10.34	6.2	5034.73	3139.09	78.91	11.08	6.54
12	110	30	25	4994	3063	78.08	12.24	7.8	4878.18	2976.47	76.65	11.42	7.66
13	110	30	25	5056	3124	78.42	11.73	8.5	4878.18	2976.47	76.65	11.42	7.66
14	100	45	20	4940	3060	79.60	15.65	9.1	5083.74	3143.80	79.92	15.13	8.39
15	100	15	30	3947	2060	73.88	13.52	9.3	4027.74	2088.60	73.26	13.17	8.88
16	120	45	20	5434	3474	75.54	12.99	8.3	5329.14	3437.00	76.15	13.00	8.52
17	110	30	25	5065	3111	76.90	11.62	7.2	4878.18	2976.47	76.65	11.42	7.66
18	100	15	20	4141	1629	77.80	15.51	9.8	4121.24	1707.20	77.88	15.52	9.69
19	100	45	30	5269	3321	72.83	13.12	6.1	5365.74	3479.70	73.02	13.21	6.33
20	110	30	25	4556	2685	76.04	12.23	8	4878.18	2976.47	76.65	11.42	7.66

X_1 - Temperature, X_2 - Time, X_3 – Moisture content, Y_{FV} - Final viscosity, Y_{SB} - Setback viscosity, Y_{GH} - Gel hardness, Y_{SP} - Swelling power, and Y_{SOL} - Solubility

Table 3C.4 Regression equations of the predicted response variables of OPT and HMT in coded values.

Responses	Regression equations in coded values	Eq. No.
OPT		
$\mathbf{Y_{FV}}$	$= +4522.57 + 122.83X_1 + 59.35X_2 + 151.53{X_1}^2 - 50.90{X_2}^2 + 74.50X_1X_2$	(3C.3)
$\mathbf{Y_{SB}}$	$= +2001.10 + 244.00X_1 - 7.50X_2 + 293.94{X_1}^2 - 239.56{X_2}^2 + 95.75X_1X_2$	(3C.4)
$\mathbf{Y_{GH}}$	$= +70.16 + 1.93X_1 + 4.47X_2 + 0.47{X_1}^2 - 1.41{X_2}^2 - 0.16X_1X_2$	(3C.5)
$\mathbf{Y_{SP}}$	$= +13.50 + 1.24X_1 - 0.072X_2 + 3.21{X_1}^2 + 0.47{X_2}^2 + 0.84X_1X_2$	(3C.6)
$\mathbf{Y_{SOL}}$	$= +10.19 + 1.95X_1 - 0.66X_2 + 0.032{X_1}^2 + 2.25{X_2}^2 - 0.70X_1X_2$	(3C.7)
HMT		
$\mathbf{Y_{FV}}$	$= +4878.18 + 301.70X_1 + 465.50X_2 + 116.50X_3 + 245.05{X_1}^2 - 308.95{X_2}^2 + 137.05{X_3}^2 - 109.63X_1X_2 + 69.37X_1X_3 + 93.87X_2X_3$	(3C.8)
$\mathbf{Y_{SB}}$	$= +2976.47 + 335.10X_1 + 470.30X_2 + 131.20X_3 + 154.32{X_1}^2 - 307.68{X_2}^2 + 116.82{X_3}^2 - 236.63X_1X_2 - 48.12X_1X_3 - 11.38X_2X_3$	(3C.9)
$\mathbf{Y_{GH}}$	$= +76.65 - 1.78X_1 + 0.33X_2 - 2.90X_3 - 3.52{X_1}^2 + 1.93{X_2}^2 - 0.83{X_3}^2 - 0.12X_1X_2 - 0.018X_1X_3 - 0.57X_2X_3$	(3C.10)
$\mathbf{Y_{SP}}$	$= +11.42 - 0.29X_1 - 0.41X_2 - 0.62X_3 + 2.55{X_1}^2 + 0.073{X_2}^2 - 0.069{X_3}^2 - 0.33X_1X_2 + 0.45X_1X_3 + 0.11X_2X_3$	(3C.11)
$\mathbf{Y_{SOL}}$	$= +7.66 - 0.81X_1 - 0.60X_2 - 1.23X_3 + 0.036{X_1}^2 - 0.51{X_2}^2 + 0.34{X_3}^2 + 0.36X_1X_2 - 0.51X_1X_3 - 0.31X_2X_3$	(3C.12)

X_1 - Temperature, X_2 - Time, X_3 – Moisture content, Y_{FV} - Final viscosity, Y_{SB} - Setback viscosity, Y_{GH} - Gel hardness, Y_{SP} - Swelling power, and Y_{SOL} - Solubility

variables. CV is the ratio of the estimate's standard deviation to the observed dependent variables' mean, which demonstrates how reproducible and repeatable the model is [36]. All the five responses for both OPT and HMT showed CV<10% in this study suggesting the possible reproducibility of the models. Based on the statistical analysis, it was observed that the developed model fitted well and good to describe the relationship between the independent variables and responses. The empirical expressions obtained by Eq. 3C.2 for prediction of the response variables for OPT and HMT are presented in the Table 3C.4; where the equations for the responses of OPT and HMT in coded values are numbered from Eq. (3C.3) to (3C.7) and from Eq. (3C.8) to (3C.12), respectively.

3C.3.3. Analysis of the model

3C.3.3.1. Pasting properties

In case of OPT, it was observed that the significant model terms for FV were the linear terms of temperature and time, quadratic term of temperature and interaction term of temperature and time with corresponding F values of 36.70, 8.57, 25.71, 9, respectively (Table 3C.5.), while in the case of HMT starch, the linear model terms of temperature and time are significant with F values of 13.77 and 32.77, respectively (Table 3C.5). The R^2 for the quadratic model was 0.920 for OPT and 0.851 for HMT indicating that the models were significant. Moreover, CV values obtained for OPT (1.09) and HMT (5.23) suggested that models were reliable and have possibility of reproducibility (CV < 10) [36]. The regression equations (3C.4) and (3C.9) in Table 3C.4 and ANOVA table (Table 3C.6) showed that linear term of temperature, quadratic terms of temperature and time in OPT, and linear terms of temperature and time, and interaction term of temperature and time in HMT were the significant model terms for SB. The response surface analysis of SB demonstrated an R^2 value of 0.899 (OPT) and 0.881 (HMT), which implied good fitting of the model. CV value for SB was observed to be 5.18 for OPT and 8.08 for HMT, which indicated that the models could be considered reproducible [20].

The response surface plots (Fig. 3C.1 and 3C.2) illustrates the effect of time and temperature on FV and SB in OPT and the effect of time, temperature and moisture content on FV and SB in HMT starches. OPT caused initial decrease, then gradual increase in FV with increasing temperature of heating (Fig. 3C.1a). Significant variation in FV was not observed when OPT treatment time was increased. SB increased with increasing temperature and time up to around 38 min (Fig. 3C.2a). Higher FV and

Table 3C.5 ANOVA for the response surface quadratic polynomial model for OPT and HMT (Final viscosity)

Source	Sum of Squares	DF	Mean Square	F Value	p-value	
Final viscosity for OPT						
Model	197436.02	5	39487.2	16.01	0.001	significant
X_1	90528.17	1	90528.17	36.7	0.0005	
X_2	21137.67	1	21137.67	8.57	0.0221	
X_1^2	63418.82	1	63418.82	25.71	0.0014	
X_2^2	7156.88	1	7156.88	2.9	0.1323	
X_1X_2	22201	1	22201	9	0.0199	
Residual	17266.77	7	2466.68			
Lack of Fit	9664.66	3	3221.55	1.7	0.3047	not significant
Pure error	7602.11	4	1900.53			
Cor Total	214702.79	12				
R^2			0.92			
CV			1.09			
Final viscosity for HMT						
Model	3782608.34	9	420289.82	6.36	0.0039	significant
X_1	910228.9	1	910228.9	13.77	0.004	
X_2	2166902.5	1	2166902.5	32.77	0.0002	
X_3	135722.5	1	135722.5	2.05	0.1825	
X_1^2	165130.01	1	165130.01	2.5	0.1451	
X_2^2	262495.51	1	262495.51	3.97	0.0743	
X_3^2	51649.01	1	51649.01	0.78	0.3976	
X_1X_2	96141.13	1	96141.13	1.45	0.2556	
X_1X_3	38503.12	1	38503.12	0.58	0.463	
X_2X_3	70500.12	1	70500.12	1.07	0.3261	
Residual	661223.41	10	66122.34			
Lack of Fit	425630.07	5	85126.01	1.81	0.266	not significant
Pure error	235593.33	5	47118.67			
Cor Total	4443831.75	19				
R^2			0.851			
CV			5.23			

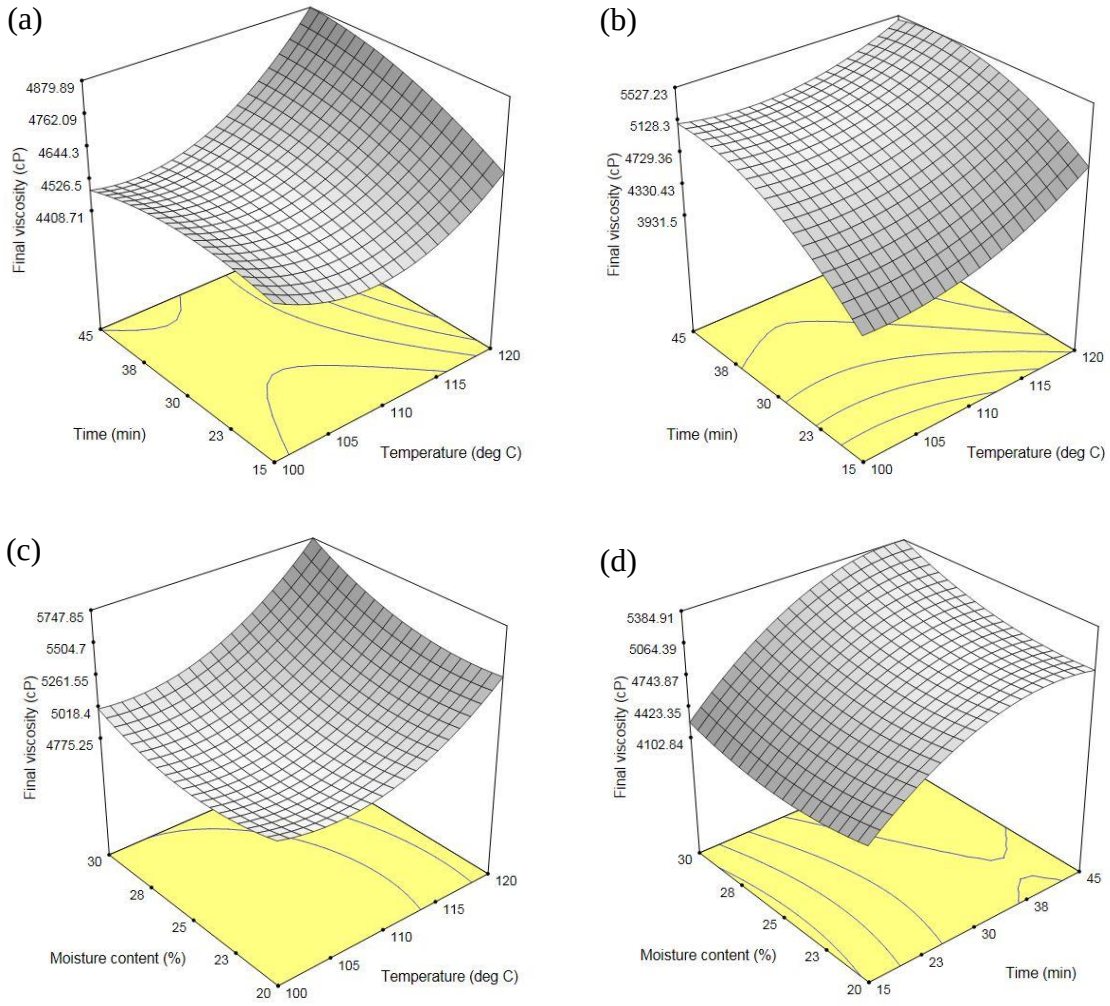

Fig. 3C.1 Response surface 3D plots: (a) Effect of time and temperature on final viscosity of OPT starch, (b-d) effect of time, temperature and moisture content on final viscosity of HMT starch.

setback due OPT may be due to the reassociation of the leached amylose fraction of starch which enhanced the gel structure during cooling. Decrease in FV and setback with increasing heating time and temperature were reported in potato starch [91], corn starch [92], and sago starch [90]. Contradictory results of decrease in FV and SB on OPT breadfruit and corn starch were reported by [73-74]. The FV value increased with increasing time and temperature within the design range in HMT whereas the increase in moisture content did not show much prominent changes in FV (Fig. 3C.1b - 3C.1d). But SB increased with increasing time, temperature and moisture content in HMT (Fig. 3C.2b - 3C.2d). However, the variation pattern of SB due to moisture content in relation to treatment time and temperature were different. Gradual increase in moisture content

Table 3C.6 ANOVA for the response surface quadratic polynomial model for OPT and HMT (setback).

Source	Sum of Squares	DF	Mean Square	F Value	p-value	
Setback for OPT						
Model	685445.8	5	137089.16	12.46	0.0022	significant
X_1	357216	1	357216	32.47	0.0007	
X_2	337.5	1	337.5	0.03	0.8659	
X_1^2	238633.92	1	238633.92	21.69	0.0023	
X_2^2	158500.2	1	158500.2	14.41	0.0068	
X_1X_2	36672.25	1	36672.25	3.33	0.1106	
Residual	77007.97	7	11001.14			
Lack of Fit	46209.17	3	15403.06	2	0.2563	not significant
Pure error	30798.8	4	7699.7			
Cor Total	762453.76	12				
R^2			0.899			
CV			5.18			
Setback for HMT						
Model	4242633.87	9	471403.76	8.25	0.0014	significant
X_1	1122920.1	1	1122920.1	19.66	0.0013	
X_2	2211820.9	1	2211820.9	38.73	< 0.0001	
X_3	172134.4	1	172134.4	3.01	0.1132	
X_1^2	65488.78	1	65488.78	1.15	0.3094	
X_2^2	260337.28	1	260337.28	4.56	0.0585	
X_3^2	37527.84	1	37527.84	0.66	0.4365	
X_1X_2	447931.13	1	447931.13	7.84	0.0188	
X_1X_3	18528.13	1	18528.13	0.32	0.5815	
X_2X_3	1035.13	1	1035.13	0.02	0.8956	
Residual	571159.33	10	57115.93			
Lack of Fit	393044	5	78608.8	2.21	0.2027	not significant
Pure error	178115.33	5	35623.07			
Cor Total	4813793.2	19				
R^2			0.881			
CV			8.08			

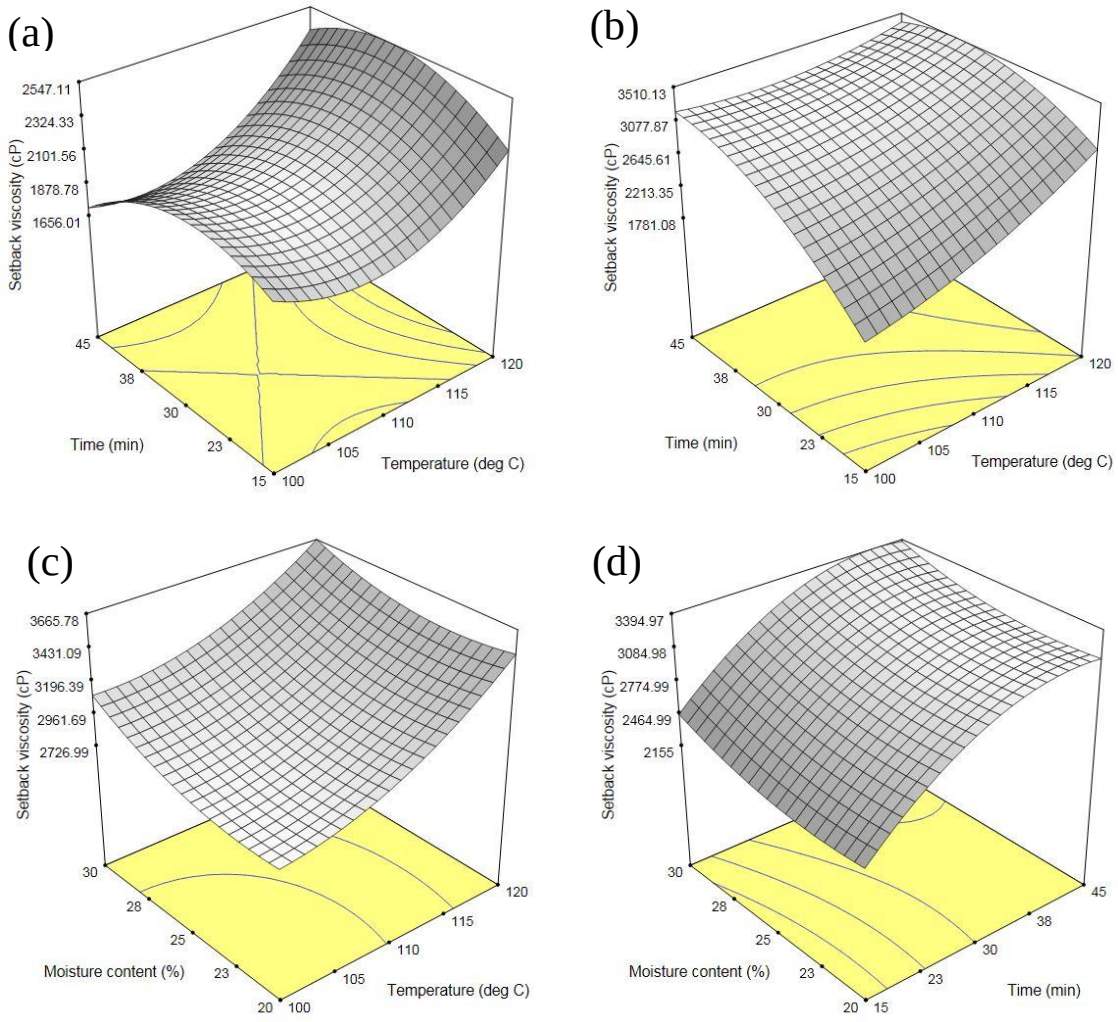

Fig. 3C.2 Response surface 3D plots: (a) Effect of time and temperature on setback of OPT starch, (b-d) effect of time, temperature and moisture content on setback of HMT starch.

and treatment time and temperature supported the polymer chain motion and enhanced the FV and SB [48]. Some authors have reported decrease in pasting properties of rice starch on HMT [6, 25]. Because of the reorientation of the starch granules and them propensity to reassociate to form a gel, the modification treatment may have tended to increase the region of crystallinity, as suggested by the rise in FV and SB following HMT [11]. HMT rice starch showing similar pattern of pasting properties have been employed for rice noodle preparation [49].

Table 3C.7 ANOVA for the response surface quadratic polynomial model for OPT and HMT (Gel hardness).

Source	Sum of Squares	DF	Mean Square	F Value	p-value	
Gel hardness for OPT						
Model	148	5	29.6	14.87	0.0013	significant
X_1	22.37	1	22.37	11.24	0.0122	
X_2	120.01	1	120.01	60.3	0.0001	
X_1^2	0.61	1	0.61	0.31	0.5957	
X_2^2	5.51	1	5.51	2.77	0.14	
X_1X_2	0.1	1	0.1	0.05	0.8314	
Residual	13.93	7	1.99			
Lack of Fit	6.27	3	2.09	1.09	0.4489	not significant
Pure error	7.66	4	1.91			
Cor Total	161.93	12				
R^2			0.914			
CV			2.02			
Gel hardness for HMT						
Model	170.4	9	18.93	13.32	0.0002	significant
X_1	31.62	1	31.62	22.25	0.0008	
X_2	1.06	1	1.06	0.75	0.4074	
X_3	83.98	1	83.98	59.1	< 0.0001	
X_1^2	34.03	1	34.03	23.95	0.0006	
X_2^2	10.29	1	10.29	7.24	0.0227	
X_3^2	1.87	1	1.87	1.32	0.2776	
X_1X_2	0.12	1	0.12	0.09	0.7758	
X_1X_3	0	1	0	0	0.9662	
X_2X_3	2.6	1	2.6	1.83	0.206	
Residual	14.21	10	1.42			
Lack of Fit	4.62	5	0.92	0.48	0.7792	not significant
Pure error	9.59	5	1.92			
Cor Total	184.61	19				
R^2			0.923			
CV			1.58			

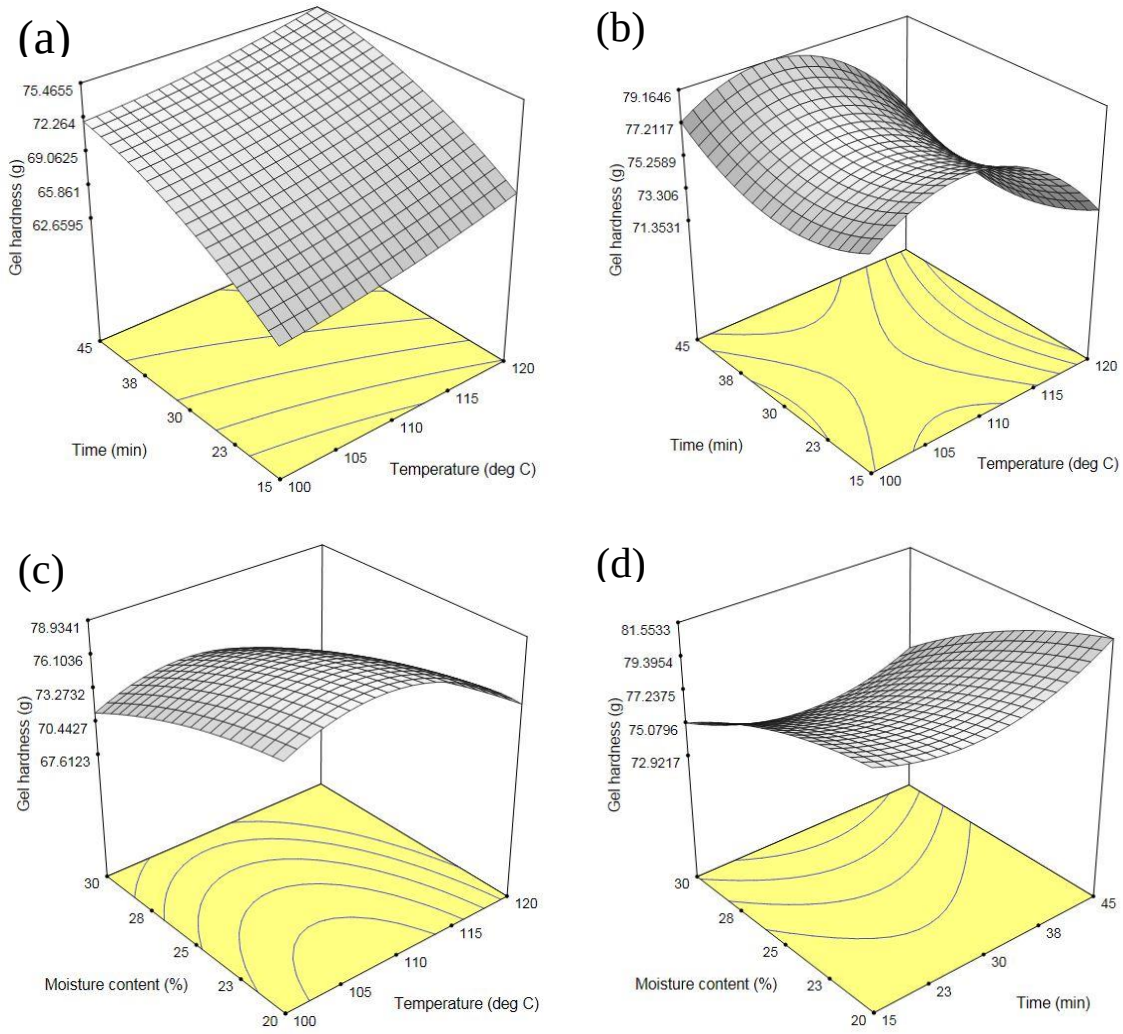

Fig. 3C.3 Response surface 3D plots: (a) Effect of time and temperature on gel hardness of OPT starch, (b-d) effect of time, temperature and moisture content on gel hardness of HMT starch.

3C.3.3.2. Gel hardness

The results in the ANOVA table (Table 3C.7) and the regression equations (3C.5) and (3C.10) in Table 3C.4 clearly illustrate that the linear terms of temperature and time for OPT and linear terms of temperature, moisture content and quadratic terms of time and temperature for HMT were significant model terms ($p < 0.05$). High R^2 value of GH in OPT (0.914) and HMT (0.923) indicated a good fit of the experimental model. Both modification methods showed high reliability of the experiments chosen for the models indicated by observed CV values of 2.02 (OPT) and 1.58 (HMT).

Gel hardness refers to the amount of force needed to deform a sample as determined by a texture analyser. OPT and HMT starches showed higher values of GH

than native rice starch (51 g). GH significantly increased with increasing time and temperature in OPT (Fig. 3C.3a). It was reported that [36] an increase in OPT reaction time favoured an increase in amylose content of starch following modification for a given amount of starch. Rice flour having higher amylose content and longer amylopectin chains exhibited harder gels because amylose is directly related to GH [98]. Starch with high final viscosity and consistency values produces gels with a harder texture [78].

Moreover, GH occurs due to the retrogradation of starch, which is related to the syneresis and crystallization of amylopectin. Time and temperature were the prominent factors affecting the GH in HMT starch (Fig. 3C.3b). Highest GH (79.94 g) was observed at the maximum time (45 min) of treatment in the design range for HMT. More perfect crystalline starch granules were formed under high temperature and moisture content [67]. With increase in moisture content the GH tended to decrease (Fig. 3C.3c - 3C.3d). Similar observation was reported in rice starch [93]. This might be due to HMT starch not expanding as much, which results in a weaker gel. Increased GH was observed during HMT of rice starch [49] and the authors ascribed it to the increased interactions between starch polymers especially in the amylose region. High GH is desirable for good quality rice noodle [13, 49, 120, 124].

3C.3.3.3. Swelling power and Solubility

It was observed from the ANOVA table (Table 3C.8) and the polynomial equations (3C.6) and (3C.11) in Table 3C.4 for SP that the linear and quadratic terms of temperature and interaction term of temperature and time are significant ($p < 0.005$) in case of OPT whereas only the linear term of moisture content and quadratic term of temperature are significant model terms in case of HMT. R^2 values for SP in OPT and HMT were 0.977 and 0.883, respectively. Moreover, the smaller values of CV < 10 for selling power in OPT (2.69) and HMT (3C.84) denoted that the experiments were responsible and reliable. For OPT, it is clearly seen from Table 4 that linear terms of temperature and time, quadratic term of time and interaction term of temperature and time were the significant model terms in case of SOL with their corresponding F values of 174.32, 19.70, 106.69 and 14.98, respectively. On the other hand, only the linear terms of independent variables were significant for SOL in HMT with high F value and $p < 0.05$ (Table 3C.9). Both the quadratic model for SOL with high R^2 values in both OPT (0.980) and HMT (0.857) showed that good relevance and interpretation of the

Table 3C.8 ANOVA for the response surface quadratic polynomial model for OPT and HMT (Swelling power).

Source	Sum of Squares	DF	Mean Square	F Value	p-value	
Swelling power for OPT						
Model	49.62	5	9.92	59.56	< 0.0001	significant
X_1	9.17	1	9.17	55.01	0.0001	
X_2	0.03	1	0.03	0.19	0.68	
X_1^2	28.38	1	28.38	170.34	< 0.0001	
X_2^2	0.61	1	0.61	3.67	0.0968	
X_1X_2	2.79	1	2.79	16.75	0.0046	
Residual	1.17	7	0.17			
Lack of Fit	0.25	3	0.08	0.36	0.7879	not significant
Pure error	0.92	4	0.23			
Cor Total	50.78	12				
R^2			0.977			
CV			2.69			
Swelling power for HMT						
Model	41.44	9	4.6	8.39	0.0013	significant
X_1	0.83	1	0.83	1.52	0.2464	
X_2	1.72	1	1.72	3.13	0.1071	
X_3	3.8	1	3.8	6.93	0.0251	
X_1^2	17.84	1	17.84	32.49	0.0002	
X_2^2	0.01	1	0.01	0.03	0.873	
X_3^2	0.01	1	0.01	0.02	0.8808	
X_1X_2	0.85	1	0.85	1.55	0.2412	
X_1X_3	1.62	1	1.62	2.94	0.117	
X_2X_3	0.09	1	0.09	0.16	0.6939	
Residual	5.49	10	0.55			
Lack of Fit	4.54	5	0.91	4.76	0.056	not significant
Pure error	0.95	5	0.19			
Cor Total	46.93	19				
R^2			0.883			
CV			5.84			

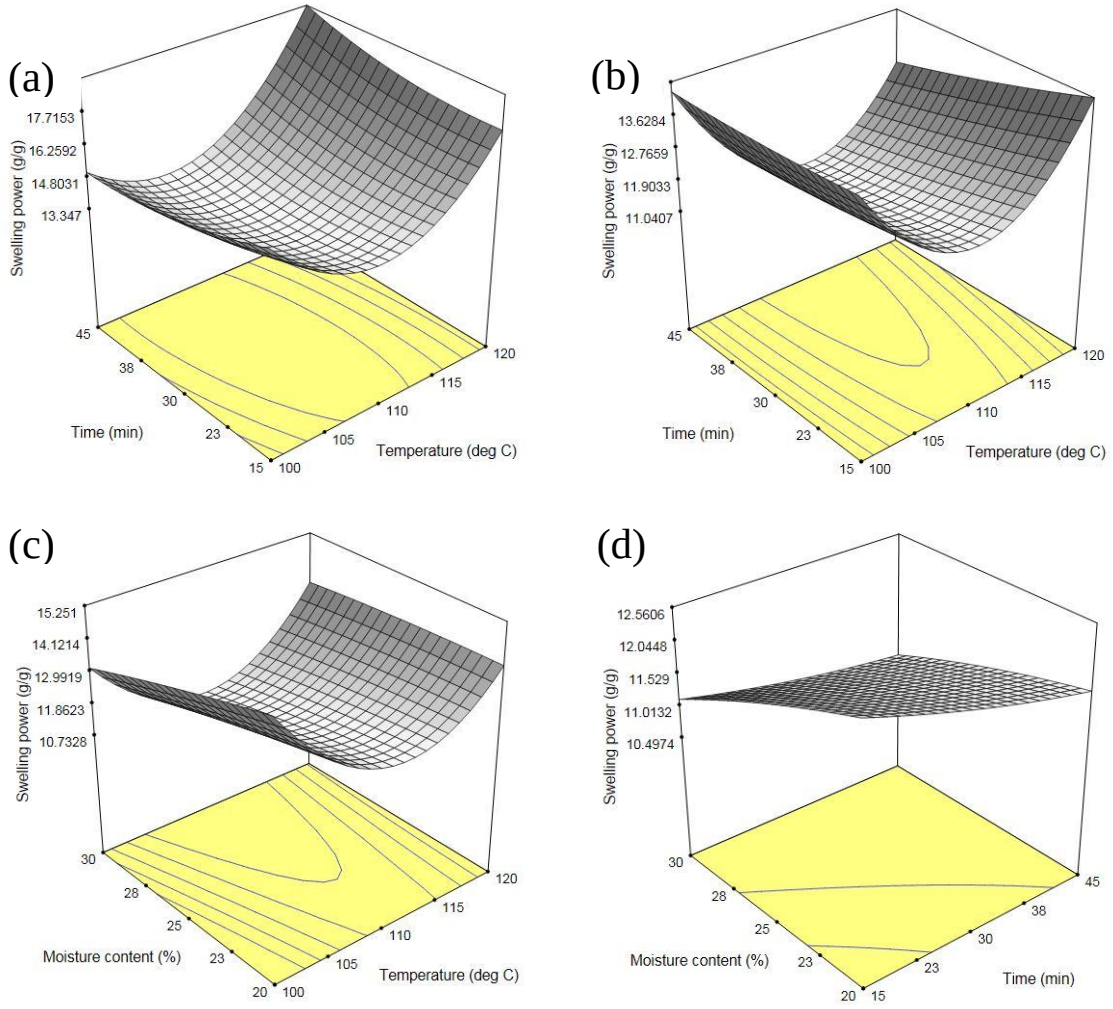

Fig. 3C.4 Response surface 3D plots: (a) Effect of time and temperature on swelling power of OPT starch, (b-d) effect of time, temperature and moisture content on swelling power of HMT starch.

model can be achieved. Meanwhile, the CV values for SOL were 3.22 and 9.35 in OPT and HMT, respectively. The degree of interaction between starch chains in the crystalline and amorphous regions is indicated by SP and SOL [58]. In OPT, SP of starch increased with increasing temperature above 110 ^{o}C but simultaneous increase in heating time decreased SP (Fig. 3C.4a), whereas OPT tended to increase SOL with increasing time and temperature (Fig. 3C.5a). These results are in agreement with the results reported elsewhere [92] in corn starch. In HMT, the effect of time and temperature showed similar trend of change in SP as in OPT (Fig. 3C.4b). SP gradually decreased when the moisture content of starch was increased from 20 to 30% with simultaneous increase in heating time (Fig. 3C.4d). Many explanations explain the decrease in SP after HMT, including rearrangement and reassociation of starch

Table 3C.9 ANOVA for the response surface quadratic polynomial model for OPT and HMT (Solubility).

Source	Sum of Squares	DF	Mean Square	F Value	p-value	
Solubility for OPT						
Model	43.87	5	8.77	67.04	< 0.0001	significant
X_1	22.81	1	22.81	174.32	< 0.0001	
X_2	2.58	1	2.58	19.7	0.003	
X_1^2	0	1	0	0.02	0.8878	
X_2^2	13.96	1	13.96	106.69	< 0.0001	
X_1X_2	1.96	1	1.96	14.98	0.0061	
Residual	0.92	7	0.13			
Lack of Fit	0.02	3	0.01	0.04	0.9895	not significant
Pure error	0.89	4	0.22			
Cor Total	44.78	12				
R^2			0.98			
CV			3.22			
Solubility for HMT						
Model	30.04	9	3.34	6.64	0.0033	significant
X_1	6.56	1	6.56	13.06	0.0047	
X_2	3.6	1	3.6	7.16	0.0232	
X_3	15.13	1	15.13	30.1	0.0003	
X_1^2	0	1	0	0.01	0.9339	
X_2^2	0.73	1	0.73	1.44	0.2572	
X_3^2	0.31	1	0.31	0.62	0.4496	
X_1X_2	1.05	1	1.05	2.09	0.1787	
X_1X_3	2.1	1	2.1	4.18	0.0681	
X_2X_3	0.78	1	0.78	1.55	0.2409	
Residual	5.03	10	0.5			
Lack of Fit	4.12	5	0.82	4.5327857	0.0614	not significant
Pure error	0.91	5	0.18			
Cor Total	35.07	19				
R^2			0.857			
CV			9.35			

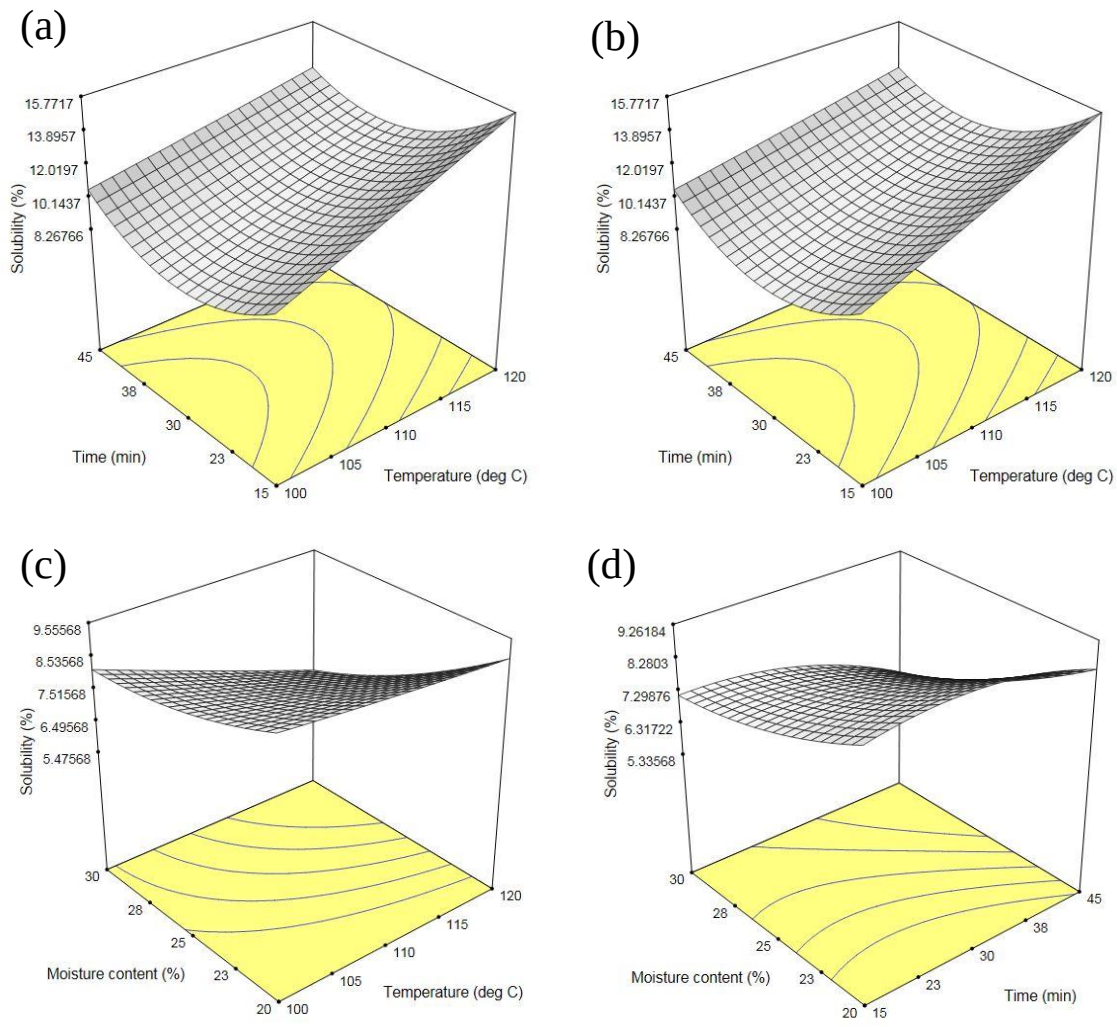

Fig. 3C.5 Response surface 3D plots showing (a) Effect of time and temperature on solubility of OPT starch, (b-d) effect of time, temperature and moisture content on solubility of HMT starch.

polymers, which slows down hydration, changes in crystallite perfection, and starch strength [27, 88, 116]. SOL was found to decrease at higher treatment time with increasing temperature (Fig. 5.5b). Moreover, it was found to decrease with increase in moisture content when time was kept constant (Fig. 5.5d) and when the temperature was increased (Fig. 5.5c). It has been reported that the unravelling of double helices during HMT decreased starch swelling and structural stability [115]. Higher moisture levels during HMT prevent amylopectin molecules in waxy maize starch from leaching out of the fragile granules, leading in a decrease in SOL [109].

3C.3.4. Optimization and validation of results

The optimum process condition was obtained by considering the independent variables within the range of experimental condition and by maximizing three response variables viz. FV, SB, GH and minimizing two response variables viz. SP and SOL. The numerical optimization solution given by the statistical software with the highest desirability was selected for both OPT and HMT. The optimum condition for OPT was a temperature of 117 ^{o}C and heating time of 35 min to give highest FV of 4718.42 cP, SB of 2312.27 cP, GH of 73.12 g and lower SP of 16.21 g/g and SOL of 11.45%. On the other hand, the optimum process condition for HMT was obtained as temperature of 111 ^{o}C, moisture content of 29% and heating time of 45 min to give highest FV of 5341.62 cP, SB of 3337.05 cP, GH of 75.11 g and SP of 10.57 g/g and SOL of 5.39%. The optimum process conditions for both OPT and HMT were experimentally validated in triplicates. The data obtained by experimental study of the optimum process conditions sufficiently agree with the predicted value generated by the optimization tool.

3C.4. Conclusion

Optimal conditions of OPT and HMT were derived for rice starch in the current investigation. RSM coupled with FCCD proved to be effective in predicting the effect of process variables for both the modification ($p<0.05$). For OPT, the optimal process condition was obtained at a temperature of 117 ^{o}C and heating time of 35 min and for HMT the optimal results were achieved at temperature of 111^{o}C, moisture content of 29% and heating time of 45 min for maximum final viscosity, setback viscosity, gel hardness, and minimum swelling power and solubility. Both the optimal treatment processes caused an increase in the final viscosity, setback viscosity and gel hardness but decreased the swelling power and solubility of the starches in both modification methods with increasing time, temperature and moisture content. Thus, as expected HMT and OPT showed substantially similar changes in the rice starch. Therefore, the study generates great scope and value for the continuation of further exploitation and application of OPT and HMT rice starches from medium broken rice of *Ranjit* variety in rice noodle making.

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