



# Effect of steaming and roasting on the quality and resistant starch of brown rice flour with high amylose content

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## ABSTRACT

The high-amylose rice cultivar Dodamssal (DDS), a potentially nutritious functional food, contains resistant starch (RS). However, its RS content varies with the processing method used. In this study, heat treatment was used to produce rice powder with enhanced RS content. The rice powder quality was improved by steaming rough rice and brown rice for 30 min and roasting the brown rice at 240 °C for 10 min. Brown rice flour (BRF) made from steamed rough rice and roasted DDS (SRRD) had higher RS content (14%), ratio of fine particle sizes (0–20 µm), and gelatinization degree (GD), as well as lower mean particle size (51 µm) and *in vitro* glycemic index than did roasted DDS. Contrastingly, BRF prepared from steamed brown rice and roasted DDS with high GD had low RS content (12%) and high mean particle size (56 µm). Hence, SRRD-BRF improves powder quality and may be beneficial for nutritious functional food production.

## 1. Introduction

Obesity can cause chronic metabolic disorders, such as coronary heart disease or diabetes, and has been reported as a disease affecting modern society (Jung, 1997). The global obesity rate has continuously increased. The association between both overweight and obesity with high cause mortality was generally consistent worldwide (Di Angelantonio et al., 2016). Due to increased awareness regarding healthcare concerns and changes in lifestyle behaviors, an increase in demand has arisen from consumers for functional, yet convenient and high-quality ingredients and convenience foods (Jung, 1997). As a staple diet for many people around the world, rice is not only consumed solely as steamed rice but is also an important ingredient in the food processing industry where rice flour can be used in various ways, including the production of rice cakes, liquid diets, desserts, and rice noodles. The

high nutrient content in brown rice has piqued the interest of consumers and has led to an increase in demand. Brown rice is more nutritious than white rice due to the presence of bran, which forms the outer layer of the rice grain. Bran contains dietary fiber, proteins, vitamin B, minerals, and antioxidants (Gul, Yousuf, Singh, & Wani, 2015).

After removing the bran layer from brown rice, the endosperm cells that remain are primarily composed of starch, which accounts for approximately 90% of milled rice dry matter (Juliano, 1985). In general, starch is hydrolyzed by enzymes, such as  $\alpha$ -amylases and glucoamylase, to yield free glucose that is absorbed in the small intestine. Alternatively, a portion of starch remains undigested (Nugent, 2005). Such unhydrolyzed starch in the small intestine becomes fermented in the large intestine and is called resistant starch (RS). As a dietary fiber and prebiotic, RS is reported to have various physiological benefits, such as colon cancer prevention, hypoglycemic effects, hypocholesterolemic

**Abbreviations:** BRF, brown rice flour; DDS, Dodamssal (*Oryza Sativa* L.); DSC, differential scanning calorimeter; eGI, estimated glycemic index; GD, gelatinization degree; HI, hydrolysis index; IM, Ilmi (*Oryza Sativa* L.); IP, Ilpumbyeo (*Oryza Sativa* L.); RDS, rapidly digestive starch; RS, resistant starch; RD, roasted DDS; SBRD, steamed brown rice and roasted DDS; SRRI, steamed rough rice and roasted Ilmi; SRRD, steamed rough rice and roasted DDS; TS, total starch; XRD, X-ray diffractometry.

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effects, and inhibition of fat accumulation by producing short-chain fatty acids (Sajilata, Singhal, & Kulkarni, 2006).

RS can be classified into the following four types according to the production method used: RS1 is the starch found in whole grains and seeds and is physically protected from enzymes; RS2 is the raw starch in foods, such as raw potatoes, unripe bananas, bean crops, and high-amylose corn, that cannot be gelatinized; RS3 is the retrograded starch formed through cooling after gelatinization and can be found in potatoes that are cooled after cooking or in processed foods like bread and cereals; RS4 is the chemically modified starch created by cross-linking chemical reagents (Nugent, 2005). As described above, there are diverse factors, including the inherent properties of natural starch formation as well as artificial thermal processing methods, which affect the formation of RS. Therefore, designing processing methods that can improve RS content is of great importance in the food industry (Sajilata et al., 2006).

Dry heat treatment results in higher RS content in various foods, including cereals, tubers, and legumes, than wet heat treatment (Platel & Shurpalekar, 1994). The gelatinization of starch granules by thermal processing is strongly influenced by the susceptibility of starch to enzymatic hydrolysis. Thus, it is reported that amylose leaches from starch granules and increases the solubility and susceptibility of starch at high moisture levels (Holm, Lundquist, Björck, Eliasson, & Asp, 1988). Meanwhile, during the roasting process, starch is damaged, partially gelatinized, and subsequently retrograded, which reportedly affects RS development (Kumar & Prasad, 2018). Steam cooking has also proven beneficial for the production of RS. Additionally, short dry pressure heating and prolonged steaming induce the formation of other types of indigestible starch in legumes or beans (Tovar & Melito, 1996). Rice can be gelatinized by steaming without adding water to the granules (Ituen & Ukpakha, 2011).

The National Institute of Crop Science in Korea has developed various functional rice plants in response to consumers' interest and demand for healthy foods. Among high-amylose rice cultivars containing RS, Goami 2, which exhibits a B-type starch crystallinity pattern, and Dodamssal (DDS), with a C-type starch crystallinity pattern, have been developed (Park, Oh, Chung, & Park, 2020). Both have relatively high protein and fat contents, high gelatinization temperatures, and round starch granule shapes. However, further development is required to process these rice cultivars, as they have unique properties distinct from those of ordinary rice.

In our previous study, we observed a more than 50% increase in the RS content in the DDS cultivar compared to that in roasted raw brown rice, but the RS content did not increase in the Ilmi (IM) cultivar, which is considered as a typical brown rice. In addition, DDS brown rice powder exhibited statistically smaller particle sizes, a lower *in vitro* estimated glycemic index (eGI), and higher RS content than did Ilpumbyeo (IP) brown rice, an intermediate-amylose rice (Park et al., 2018, 2019). Therefore, given the known health functions of roasted DDS brown rice as a food product and the potential benefits of processing it into a powder, further study is necessary. Accordingly, in the current study, DDS brown rice and DDS rough rice were separately treated by steaming and roasting after drying, as a pretreatment to improve the quality of the powdered raw material. Roasted DDS and roasted IM, which was steamed in advance, were used as control samples. Starch appearance, particle size, crystal structure, thermal properties, RS content, and digestibility of the treated brown rice flours were analyzed to obtain preliminary data regarding their applicability for use in convenience food and for the establishment of a manufacturing process.

## 2. Materials and methods

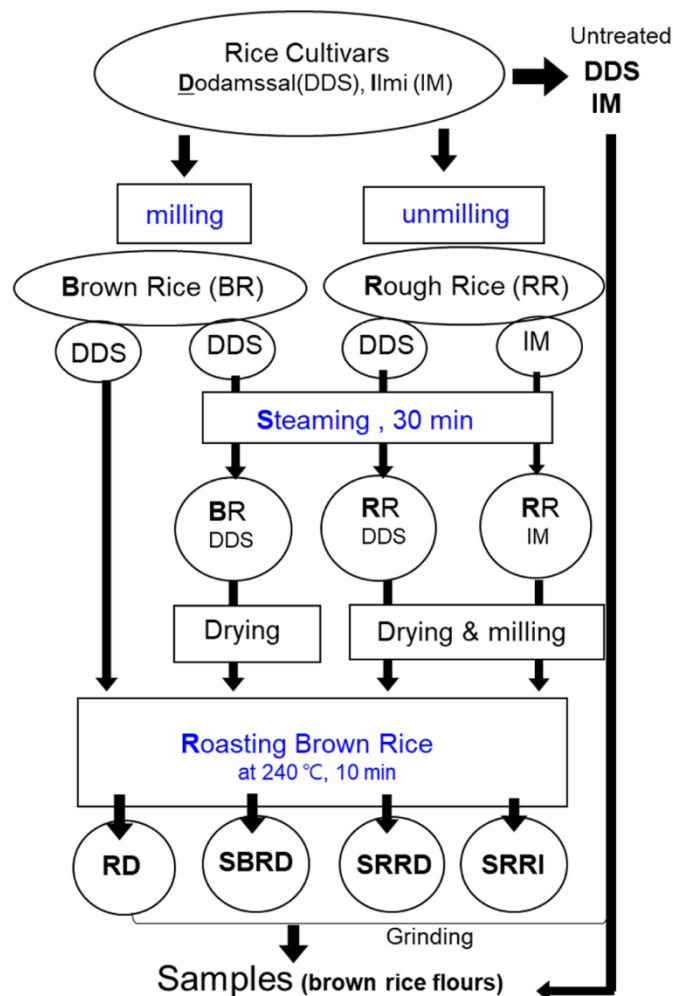
### 2.1. Materials

The rice cultivars DDS and IM were produced in 2017 by the National

Institute of Crop Science (Suwon, Gyeonggi-do, South Korea). Their polished rice amylose levels were 40.6% and 20.6%, respectively (Park et al., 2020). Harvested mature seeds were air-dried for 1 d. A portion of the seeds was milled into brown rice using a sheller (Model SY88-TH; SsangYong Motor Co. Ltd., Incheon, Korea), while unmilled rice was used for steaming or roasting (heat treatment). The distilled water and ethanol used were of HPLC grade.

### 2.2. Sample preparation

Six samples were prepared by heat treatment (Fig. 1). The first sample comprised DDS (RD) that was roasted at 240 °C for 10 min; these conditions were previously established to yield a high RS content in DDS brown rice flour (BRF) and to lead to a low *in vitro* eGI and high palatability (Park et al., 2018). Herein, the samples were heat-treated with a far-infrared roaster (Model FEC-006; Biotech, Incheon, Korea). All roasting was performed under the same conditions. The second sample consisted of steamed brown rice and roasted DDS (SBRD) obtained by steaming 1000 g of DDS brown rice at 95 °C for 30 min, drying at 40 °C after washing briefly, and roasting at 240 °C for 10 min. The third sample was steamed rough rice and roasted DDS (SRRD) obtained by steaming, washing, drying, milling, and roasting 1000 g of DDS rough rice. The fourth sample was steamed rough rice and roasted Ilmi (SRRI)



**Fig. 1.** Sample (brown rice flours) preparation. RD, roasted Dodamssal; SBRD, steamed brown rice and roasted Dodamssal; SRRD, steamed rough rice and roasted Dodamssal; SRRI, steamed rough rice and roasted Ilmi; DDS, Untreated Dodamssal; IM, Untreated Ilmi. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

prepared by steaming, washing, drying, milling, and roasting plain rough rice in the same method as that of SRRD. The samples were prepared in the same manner as heat-treated BRF. To evaluate gelatinization, untreated DDS and IM were pulverized in the same way as the heat-treated BRF and used as controls. They were pulverized in a commercial grain grinder (Stenis Versatile Pulverizer; Yuseong Food Machinery Co. Ltd., Daegu, Korea), filtered through a 160-mesh standard sieve, and analyzed.

### 2.3. Morphology of heat-treated brown rice flour

The four heated and milled BRF samples were gold-coated to increase their conductivity, so that their particle sizes could be evaluated under a scanning electron microscope (SEM-3000; Hitachi Ltd., Tokyo, Japan) at 2000× magnification.

### 2.4. Particle size measurement of heat-treated brown rice flour

The particle size distribution of heat-treated and milled brown rice was determined using a particle size analyzer (Mastersizer 2000; Malvern Instruments Ltd., Malvern, UK) using ethanol as the solvent.

### 2.5. X-ray diffractometry (XRD) of heat-treated brown rice flour

XRD analyses were conducted using an X-ray diffractometer (D8 ADVANCE with DAVINCI; Bruker, Hamburg, Germany). The detector used was LYNXEYE XE, the generator was operated at 40 kV, 40 mA, and (2θ) 5–40°, the scanning speed was 2.0 s/step, and the wavelength (λ) Cu Kα1 was 1.5418 Å.

### 2.6. Gelatinization properties

The thermal properties of heat-treated BRF were determined using a differential scanning calorimeter (DSC) (DSC Q1000; TA Instruments Inc., New Castle, DE, USA). Nine milligrams of BRF sample were accurately weighed, mixed with 21 mg of distilled water, and hermetically sealed in high volume pans (TA Instruments Inc., New Castle, DE, USA). The samples were equilibrated overnight to room temperature, and the pan was heated from 20 °C to 150 °C at a rate of 10 °C/min. An empty aluminum pan served as a reference. Data were calculated using the endotherm plot in the DSC software. The gelatinization degree (GD) was calculated using Eq. (1) (Błaszczak et al., 2007).

$$GD = \{ (H_{ns} - H_{ts}) / H_{ns} \} \times 100\% \quad (1)$$

where,  $H_{ns}$  and  $H_{ts}$  are the melting enthalpies of raw and heat-treated BRF, respectively.

### 2.7. RS content

The RS content was measured and compared among the six samples. To this end, an RS assay kit (Megazyme International Ireland Ltd., Wicklow, Ireland) and the Association of Official Agricultural Chemists (AOAC) method were used. Pancreatin α-amylase was added to a 100 mg sample, and the mixture was incubated at 37 °C for 16 h. The precipitate was dispersed and dissolved in 2 M KOH. Subsequently, 1.2 M sodium acetate buffer (pH 3.8) and amyloglucosidase were added and the mixture was incubated at 50 °C for 30 min. The RS content was converted to D-glucose based on the amount of starch hydrolyzed. The changes in moisture content after the various heat treatment conditions were corrected to estimate the RS content.

### 2.8. Starch digestibility of heat-treated brown rice flour

Starch digestibility was determined according to a previous study (Park et al., 2018). The starch digestibility value was used to determine

the hydrolysis index (HI) and eGI of heat-treated BRF. To calculate BRF starch digestibility, the total starch content (TS) in BRF was determined using a megazyme total starch assay kit (Megazyme International Ireland Ltd.). Briefly, porcine pancreatic α-amylase (P7545; Sigma-Aldrich Corp., St. Louis, MO, USA) was dispersed in distilled water. The mixture was centrifuged at 1500 × g for 10 min, and the supernatant was isolated and mixed with 0.3 mL amyloglucosidase (A9913; Sigma-Aldrich Corp.). Subsequently, 4 mL of sodium acetate buffer (pH 5.2) was added to a 100 mg sample. One milliliter of the enzyme mixture and five glass beads were combined and stirred at 150 rpm. After 180 min, the reaction product was mixed with 80% (v/v) ethanol solution. The glucose content was determined using glucose oxidase and peroxidase assay kits (Megazyme International Ireland Ltd.). The TS content and the ratio of the areas of the rice flour and standard material (white bread) digestibility curves were used to calculate the HI. The eGI was obtained from the equation ( $eGI = 39.71 + 0.549 HI$ ).

### 2.9. Statistical analysis

All data are presented as means of triplicate measurements and were analyzed with SAS v. 9.2 (SAS Institute Inc., Cary, NC, USA). Statistical significance was analyzed by one-way ANOVA and Duncan's multiple comparison.  $P < 0.05$  indicated statistically significant differences between treatment means.

## 3. Results and discussion

### 3.1. Morphology of heat-treated brown rice flours

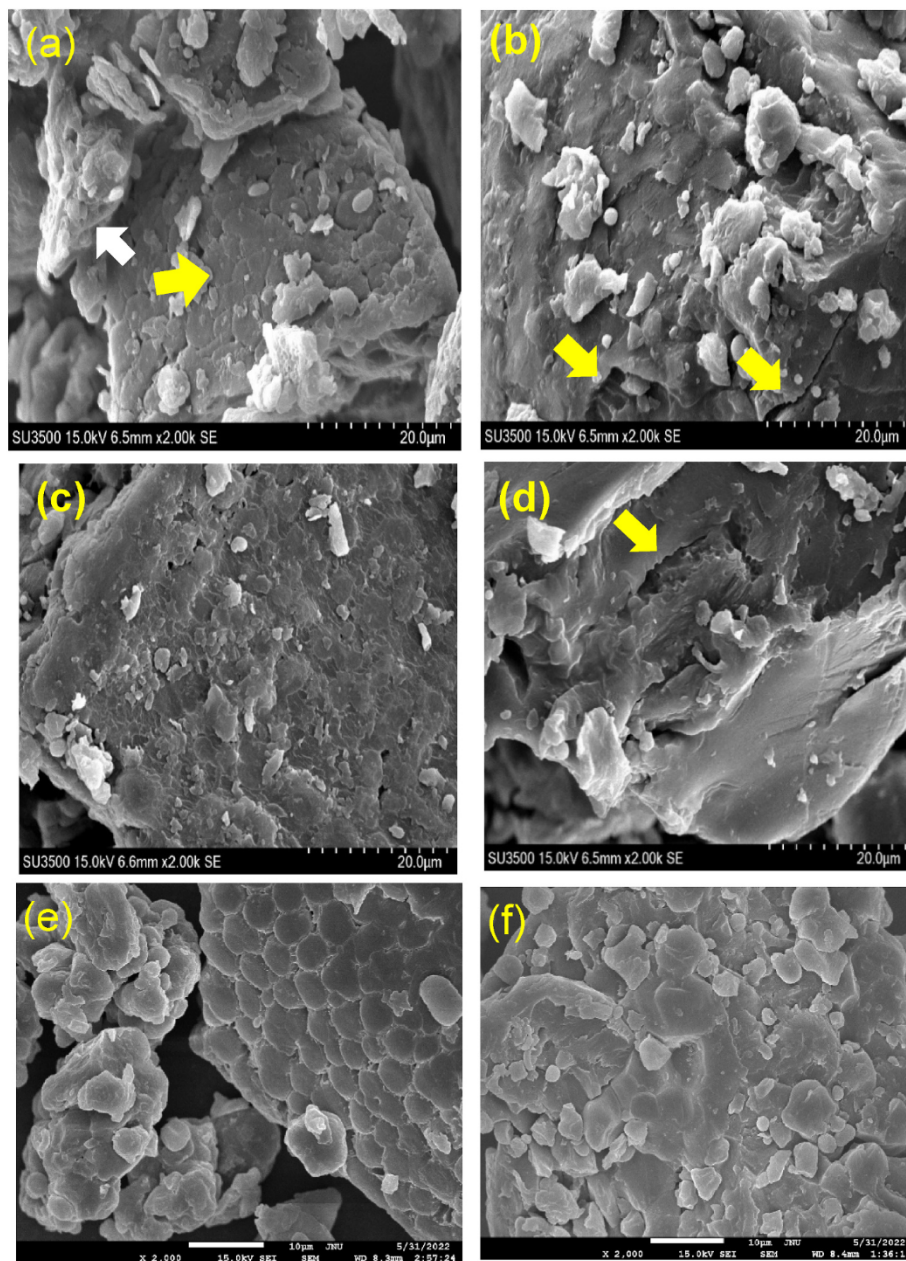
The SEM images of the four heat-treated BRF samples observed at 2000× are depicted in Fig. 2. The RD appeared as large lumps formed by partial gelatinization and subsequent granule aggregation. Smaller, irregular, and amorphous granules were detected in the RD (Fig. 2a). We observed round starch granules similar to those in raw starch (Fig. 2e). The starch granules had coalesced due to roasting. Nevertheless, their boundaries were easily distinguishable, and empty spaces were detected between them. The large lumps seen in the other samples (Fig. 2b,c, and 2d) were ≥40–50 μm in diameter and were the result of gelatinization. Granules of various sizes and shapes were observed and resembled those in the RD. The SBRD surface was uneven, and the starch granules had no distinct shapes or boundaries (Fig. 2b). However, they did present with long fissures and narrow cracks (arrows). SRRD also had empty spaces similar to those in the RD; however, the granules were amorphous. The overall surface of SRRD was rougher than that of SBRD (Fig. 2c). SRRI had the smoothest and the flattest surface. Nevertheless, it also had long empty spaces resembling those in SBRD and fissures shaped like concave angular granules 1–2 μm in diameter (Fig. 2d).

The granules expanded and their structure was disrupted when the heating temperature rose during gelatinization or the starch particles absorbed sufficient moisture. However, if water cannot penetrate the starch granules, only the outer granule layers become hydrated (Gul et al., 2015). In the present study, the shapes of the starch particles could be observed in the RD without steam treatment. In contrast, the SBRD subjected to steam treatment after milling was far more exposed to moisture and heat than the SRRD surrounded by rice husk. Hence, the former lost its original structure.

### 3.2. Particle size distribution of heat-treated brown rice flours

The particle size distribution of heat-treated BRF is shown in Table 1. RD and SRRD showed a high, slightly narrow particle size distribution. However, particle size distribution for SBRD was relatively wider, whereas that for SRRI was shifted toward the right (graph not shown). The distribution ratio of particles >150 μm in diameter was the highest for SRRI followed by SBRD (Table 1). The median particle diameter





**Fig. 2.** Scanning electron micrographs of heat-treated brown rice flour at 2000 $\times$ . (a) Roasted Dodamssal (RD), (b) steamed brown rice and roasted Dodamssal (SBRD), (c) steamed rough rice and roasted Dodamssal (SRRD), (d) steamed rough rice and roasted Ilmi (SRRI). DDS, Untreated Dodamssal; IM, Untreated Ilmi. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

RD appeared as large lumps formed by partial gelatinization and subsequent granule aggregation (yellow arrow). Smaller, irregular, amorphous granules were detected in the RD (white arrow in 2a). SBRD and SRRI granules presented with long fissures and narrow cracks (yellow arrow in 2b and 2d).

**Table 1**

Particle size distributions and median diameters of particles of heat-treated brown rice flours.

Samples	Particle size distribution (%)								Median diameter ( $\mu\text{m}$ )
	0–20 ( $\mu\text{m}$ )	20–40 ( $\mu\text{m}$ )	40–60 ( $\mu\text{m}$ )	60–100 ( $\mu\text{m}$ )	100–150 ( $\mu\text{m}$ )	150–200 ( $\mu\text{m}$ )	200–300 ( $\mu\text{m}$ )	300–400 ( $\mu\text{m}$ )	
DDS	19.0 $\pm$ 0.3 <sup>c</sup>	22.2 $\pm$ 0.1 <sup>a</sup>	14.3 $\pm$ 0.2 <sup>a</sup>	25.0 $\pm$ 0.3 <sup>a</sup>	11.4 $\pm$ 0.2 <sup>c</sup>	5.6 $\pm$ 0.3	0.7 $\pm$ 0.1 <sup>d</sup>	n.d.	53.5 $\pm$ 0.5 <sup>d</sup>
RD	20.9 $\pm$ 0.2 <sup>b</sup>	22.4 $\pm$ 0.1 <sup>a</sup>	14.3 $\pm$ 0.1 <sup>a</sup>	25.0 $\pm$ 0.2 <sup>a</sup>	11.1 $\pm$ 0.1 <sup>c</sup>	5.6 $\pm$ 0.1 <sup>c</sup>	0.7 $\pm$ 0.1 <sup>d</sup>	n.d.	53.0 $\pm$ 0.4 <sup>d</sup>
SBRD	20.6 $\pm$ 0.3 <sup>b</sup>	21.8 $\pm$ 0.3 <sup>b</sup>	11.8 $\pm$ 0.2 <sup>c</sup>	19.9 $\pm$ 0.3 <sup>d</sup>	10.8 $\pm$ 0.1 <sup>d</sup>	8.6 $\pm$ 0.3 <sup>b</sup>	5.1 $\pm$ 0.5 <sup>b</sup>	1.4 $\pm$ 0.2 <sup>b</sup>	56.0 $\pm$ 1.3 <sup>c</sup>
SRRD	21.6 $\pm$ 0.4 <sup>a</sup>	22.7 $\pm$ 0.2 <sup>a</sup>	14.2 $\pm$ 0.2 <sup>a</sup>	24.6 $\pm$ 0.2 <sup>b</sup>	11.0 $\pm$ 0.2 <sup>c,d</sup>	5.5 $\pm$ 0.2 <sup>c</sup>	0.5 $\pm$ 0.1 <sup>d</sup>	n.d.	51.7 $\pm$ 0.8 <sup>d</sup>
SRRI	16.6 $\pm$ 0.2 <sup>d</sup>	18.2 $\pm$ 0.3 <sup>c</sup>	11.5 $\pm$ 0.2 <sup>d</sup>	22.8 $\pm$ 0.2 <sup>c</sup>	13.1 $\pm$ 0.2 <sup>a</sup>	10.1 $\pm$ 0.3 <sup>a</sup>	6.0 $\pm$ 0.3 <sup>a</sup>	1.6 $\pm$ 0.1 <sup>a</sup>	69.7 $\pm$ 1.4 <sup>a</sup>
IM	18.9 $\pm$ 0.2 <sup>c</sup>	21.2 $\pm$ 0.4 <sup>b</sup>	12.6 $\pm$ 0.2 <sup>b</sup>	23.9 $\pm$ 0.5 <sup>b</sup>	12.3 $\pm$ 0.2 <sup>b</sup>	7.7 $\pm$ 0.3	3.2 $\pm$ 0.1 <sup>c</sup>	0.3 $\pm$ 0.1	59.7 $\pm$ 0.9 <sup>b</sup>

<sup>a–d</sup> Values with different letters within a column are significantly different ( $P < 0.05$ ) as determined using Duncan's multiple range test. Values represent the mean of three independent experiments.

DDS, untreated Dodamssal; RD, roasted Dodamssal; SBRD, steamed brown rice and roasted Dodamssal; SRRD, steamed rough rice and roasted Dodamssal; SRRI, steamed rough rice and roasted Ilmi; IM, untreated Ilmi; n.d., not detected.

value was the highest for SRRI and the lowest for SRRD. The distribution ratio of particles with a diameter <150  $\mu\text{m}$  was low for SBRD. In contrast, the distribution ratios of the particles with diameters between those of RD and SRRD were not significantly different except for those <20  $\mu\text{m}$  in diameter.

Granule surface roughness may increase with decreasing rice flour granule size. A non-basmati rice variety has rougher particles than a basmati variety due to the presence of fine particles (Jan, Karde, Ghoroi, & Saxena, 2018). Meanwhile, another study reported that heat moist treatment showed that moisture and thermal energy caused partial gelatinization of starch, resulting in a slight roughness of the granule surface (Wang et al., 2018). In the present study, the SRRD particles had the highest distribution ratio in the finest 0–20- $\mu\text{m}$  diameter range and the roughest surfaces (Fig. 2C, Table 1). SRRD possibly showed a rough surface because it was less gelatinized than SBRD and SRRI (Table 2).

### 3.3. XRD of heat-treated brown rice flours

The XRD analyses of the four heat-treated BRF samples are shown in Fig. 3a. The crystalline structure of the BRF derived from untreated IM showed intense peaks at 15° 2 $\theta$  and 23° 2 $\theta$ , two inseparable peaks at 17–18° 2 $\theta$ , and a very weak peak at 20° 2 $\theta$ . In contrast, the crystalline structure of the DDS BRF (Fig. 3a gray dotted line) showed peaks at 5° 2 $\theta$ , 22° 2 $\theta$ , and 24° 2 $\theta$ , 20° 2 $\theta$ , and a strong continuous peak at 17° 2 $\theta$ . The foregoing indicated a crystal structure of type B. The treated DDS had a similar XRD pattern as that of the untreated DDS, except that the former exhibited weaker peaks. The order of peak strength was RD > SRRD > SBRD. After DDS heat treatment, the amylose-lipid peak at 20° 2 $\theta$  intensity was comparatively lower. However, a peak at 20° 2 $\theta$  was observed for SRRI subjected to heat treatment. Moreover, it displayed the strongest peak intensity.

DDS starch presented with a C-type crystalline structure, which is a combination of A- and B-type crystalline structures. In addition, it showed unseparated peaks at 17° 2 $\theta$  and 23° 2 $\theta$ , with an ~31% crystallinity. However, DDS BRF (Fig. 3a.) revealed a different crystal pattern at 22–24° 2 $\theta$  resembling that of the polished DDS flour mentioned in a previous study (Park et al., 2020). Table 2 indicates that the crystallinity of BRF was lower than that of DDS starch. A previous study stated that the crystallinity of rice flour was lower than that of rice starch (Mir & Bosco, 2014).

The amylose-lipid peaks at 20° 2 $\theta$  intensified with heat treatment, were V-type, and reflected the combination of amylose and lipid (Fig. 3a.). A similar result was reported in a previous study (Nasir-ahmadi, Abbaspour-Fard, Emadi, & Khazaei, 2014). The peaks were weak for raw flour, but increased with heat treatment, as the latter increased the moisture content. Over time, lipids form inclusion complexes with the amylose leached from starch granules during cooking (Pan et al., 2017). The loss of the A-type crystal pattern and the formation of a weak peak comprising A- and V-type crystal patterns were reported for steam-treated parboiled brown rice (Wahengbam & Hazarika, 2019). SRRI had a higher GD than did other samples. The peak

intensity decreased at 17–18° 2 $\theta$ , but the V-type peak at 20° 2 $\theta$  intensity increased. The XRD for the heat-treated high-amylose DDS (RD, SBRD, and SRRD) showed a stronger peak at 20° 2 $\theta$  than did that for intermediate-amylose cultivar SRRI. For RD, SBRD, and SRRD, the 20° 2 $\theta$  peak strength increased with increasing GD. This finding is consistent with that of an earlier report (Rattanamechaikul, Soponronnarit, & Prachayawarakorn, 2014). Peak intensities differed among samples due to peak loss and increase in intensity caused by the heat treatment. Nevertheless, there were no significant differences among samples in terms of relative crystallinity.

### 3.4. Gelatinization properties of heat-treated brown rice flours

DSC thermograms show the thermal properties of the four heat-treated BRF samples (Fig. 3b). SBRD and SRRI exhibited a single endothermic transition. RD and SRRD showed their first peaks (P) at a low temperature and their second peaks (M1) at a high temperature. Hence, their endothermic transitions were biphasic. Gelatinization temperature, enthalpy, and degree are shown in Table 2. Relative to those for untreated DDS, the onset temperature ( $T_o$ ) and the main peak temperature ( $T_p$ ) of RD and SRRD decreased from 68.9 °C to 78.6 °C to 64.2–64.3 °C and 70.2–70.6 °C, respectively (Fig. 3b and Table 2). SBRD had the highest  $T_o$ ,  $T_p$ , and  $\Delta H_P$ . In the order of SRRI, SBRD, SRRD, and RD,  $\Delta H_T$  showed values of 1.3, 1.4, 1.8, and 2.0 J/g, respectively, and GD showed values of 51.2, 48.0, 35.1, and 27.9%, respectively. These differences were statistically significant ( $P < 0.05$ ).

Donovan (1979) named an endothermic transition, which occurs when the moisture content is >66% or the water/starch ratio is > 1.5, located between 60 °C and 80 °C in the DSC profile as endotherm G. Moreover, another endothermic transition, designated endotherm M1, occurs at a second-high temperature and reduced moisture. Hence, M1 endotherm occurs in starch with a moderate moisture level range of 34–66% (Collar, Jiménez, Conte, & Piga, 2015). Biphasic endotherms occur in a limited moisture-starch system (S. Wang & Copeland, 2013) and have been explained by various proposed models. The G endotherm indicates plasticization in amorphous regions, crystalline structure breakdown caused by starch expansion, and melting of unstable crystal regions in insufficient water. In contrast, the M1 endotherm represents the presence of less hydrated regions or the melting of a more stable crystalline structure. Even with sufficient water (starch: moisture = 1:10), however, a biphasic transition may be observed (Xing, Li, Wang, & Adhikari, 2017), and Vanier et al. (2012) reported that the second peak observed after long hydrothermal treatments, such as heat moisture treatment, may be explained by the presence of two crystalline structures with different thermal stabilities. The ratio of moisture to BRF was 2.23 for all samples. Thus, the second peak is explained by starch rearrangement in response to heating, as well as by the observed gelatinization properties. However, the moisture content during gelatinization of DSC analysis does not account for the second peak. Fig. 3a shows second peaks in RD and SRRD as their GDs were relatively lower than those of SRRI and SBRD. Therefore, heat-treated starch and raw starch

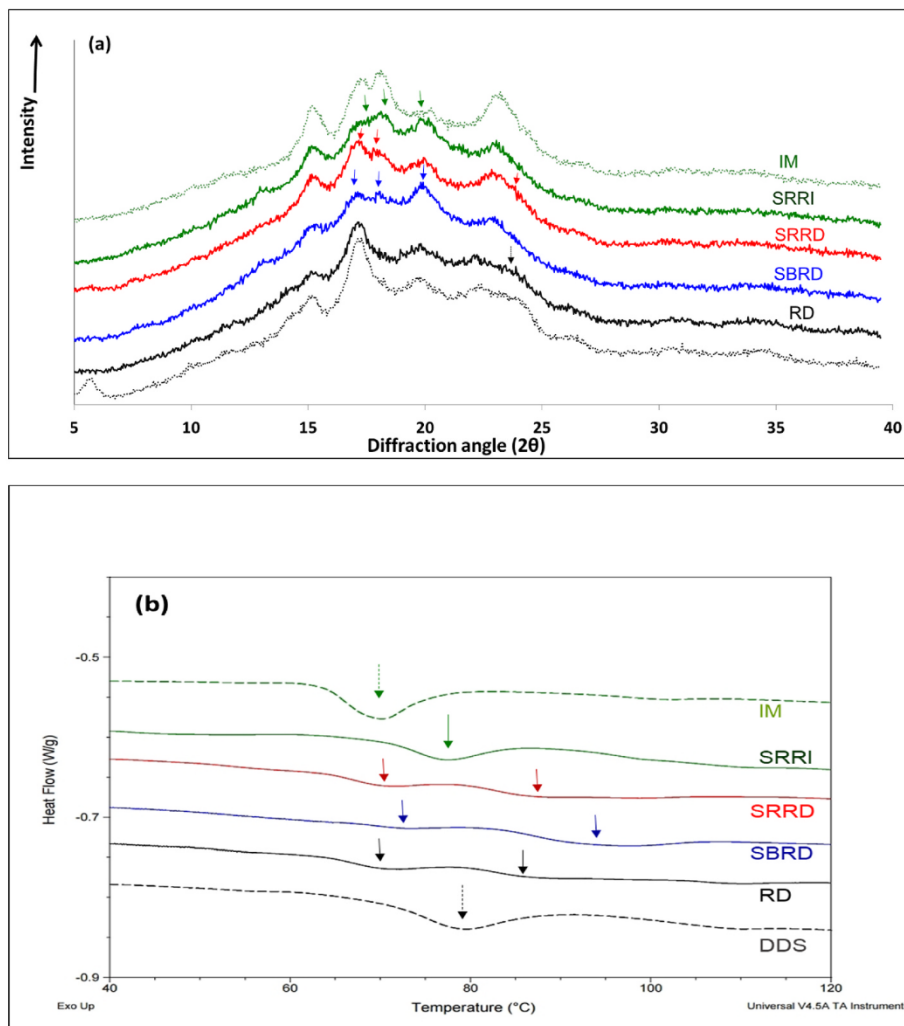
**Table 2**  
Resistant starch content, crystallinity, and thermal properties of heat-treated brown rice flours.

Samples	RS (%)	X <sub>c</sub> (%)	T <sub>o</sub> (°C)	T <sub>p</sub> (°C)	T <sub>M1</sub> (°C)	$\Delta H_P$ (J/g)	$\Delta H_{M1}$ (J/g)	$\Delta H_T$ (J/g)	GD (%)
DDS	8.6 ± 0.5 <sup>d</sup>	28.3 ± 0.5 <sup>b</sup>	69.0 ± 0.6 <sup>b</sup>	78.7 ± 0.8 <sup>b</sup>	–	–	–	2.6 ± 0.3 <sup>a</sup>	–
RD	13.0 ± 0.1 <sup>b</sup>	27.3 ± 0.5 <sup>b</sup>	64.3 ± 0.1 <sup>c</sup>	70.6 ± 0.4 <sup>d</sup>	87.5 ± 0.2 <sup>ns</sup>	0.7 ± 0.0 <sup>c</sup>	1.3 ± 0.4 <sup>ns</sup>	2.0 ± 0.1 <sup>b</sup>	27.9 <sup>d</sup>
SBRD	12.1 ± 0.1 <sup>c</sup>	27.7 ± 1.5 <sup>b</sup>	82.8 ± 0.1 <sup>a</sup>	93.7 ± 0.6 <sup>a</sup>	–	1.8 ± 0.1 <sup>a</sup>	–	1.4 ± 0.0 <sup>d</sup>	48.0 <sup>b</sup>
SRRD	14.0 ± 0.1 <sup>a</sup>	26.1 ± 0.7 <sup>b</sup>	64.2 ± 0.6 <sup>c</sup>	70.2 ± 0.3 <sup>d</sup>	87.4 ± 0.3 <sup>ns</sup>	0.7 ± 0.0 <sup>c</sup>	1.2 ± 0.2 <sup>ns</sup>	1.8 ± 0.1 <sup>c</sup>	35.1 <sup>c</sup>
SRRI	0.6 ± 0.1 <sup>e</sup>	26.3 ± 0.5 <sup>b</sup>	71.3 ± 0.7 <sup>b</sup>	76.8 ± 0.4 <sup>c</sup>	–	1.3 ± 0.3 <sup>b</sup>	–	1.3 ± 0.0 <sup>e</sup>	51.2 <sup>a</sup>
IM	0.5 ± 0.1 <sup>e</sup>	30.5 ± 0.6 <sup>a</sup>	63.4 ± 0.3 <sup>c</sup>	69.8 ± 0.1 <sup>d</sup>	–	–	–	2.5 ± 0.2 <sup>a</sup>	–

<sup>a–e</sup> Values with different letters within a column are significantly different ( $P < 0.05$ ) as determined by Duncan's multiple range test.

<sup>ns</sup> means Not Statistically Significant. Values represent means of three independent experiments. RS, resistant starch; X<sub>c</sub>, crystallinity; T<sub>o</sub>, onset temperature; T<sub>p</sub>, main peak temperature; T<sub>M1</sub>, minor transition temperature; H<sub>p</sub>, enthalpy of main endotherm; H<sub>M1</sub>, enthalpy of minor endotherm; H<sub>T</sub>, enthalpy of total endotherm; GD, degree of gelatinization; DDS, untreated Dodamssal; RD, roasted Dodamssal; SBRD, steamed brown rice and roasted Dodamssal; SRRD, steamed rough rice and roasted Dodamssal; SRRI, steamed rough rice and roasted Ilmi; IM, untreated Ilmi.





**Fig. 3.** X-ray diffraction pattern (a), and differential scanning calorimeter thermograms (b) for heat-treated brown rice flour. In 3b, the 20° 2θ peak intensity of SRRD and SBRD was higher than the others (green and blue arrows at 20° 2θ). As the 24° 2θ peak, which represents the B-type starch characteristic of DDS, weakens, it changes into the form of A-type starch. Therefore, it can be observed only in SRRD and RD (red and black arrows, respectively, at 24° 2θ). The one large peak at 17 °C of DDS is divided during heat treatment and changes into the characteristics of SRRD A type rice. Two weak peaks are observed in SRRD and SBRD (red and blue arrows at 17 and 18° 2θ, respectively). RD (roasted Dodamssal); SBRD (steamed brown rice and roasted Dodamssal); SRRD (steamed rough rice and roasted Dodamssal); SRRI (steamed rough rice and roasted Ilmi); DDS (untreated Dodamssal BRF); IM (untreated Ilmi BRF). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

have different structures. RD was only roasted, and SRRD was minimally exposed to moisture during steaming due to its intact hull. Both would have far more unhydrated regions than SBRD and would undergo M1 transition during melting.

The destruction and rearrangement of starch during hydrothermal treatment influence the changes in the melting temperature (peak) and shift the endothermic transition to higher temperatures via amylose-amylose, amylose-amylopectin, and amylose-lipid interactions (Jiranuntakul, Puttanlek, Rungsardthong, Pancha-Arnon, & Uttapap, 2011). In the present study, SBRD had the highest  $T_p$  as it also had an intense V-type peak (20° 2θ) caused by an amylose-lipid interaction. SBRD had a higher  $T_p$  than did SRRI possibly because of the amylose-amylose interactions in response to the heat treatment and the high ratio of long chains in the DDS cultivar (Table 2 and Fig. 3a).

Jörgen Holm, Björck, and Eliasson (1988) reported a GD range of 22–65% when starch was gelatinized during roasting. Table 2 discloses a GD of 27.9% after a single roasting treatment. Moreover, an extra treatment (steam) had a synergistic effect on GD increase.

The particle size distribution is explained by differences in hardness among rice grain cultivars. Thus, a low hardness correlates with brittleness and fine granules (Jan et al., 2018). In addition, gelatinization, hardness, and fracture resistance increase with the steaming time. Hardness is an important physical property of parboiled rice consumed as cooked rice because it augments fracture resistance during milling. Here, GD was the highest for SRRI followed by SBRD (Table 2), as their median particle diameters were large. This finding is consistent with

that of a previous study (Taghinezhad, Khoshtaghaza, Minaei, Suzuki, & Brenner, 2016). However, increased gelatinization and powder particle size may hinder the development of powdered products after heat treatment.

SRRI had higher GD than did SRRD. The observed morphological difference between the SRRD and SRRI granules under the same heat treatment (Fig. 2c and d) may be explained by the fact that their respective GDs were 35.1 and 51.2. In Table 2, this difference was statistically significant ( $P < 0.05$ ).

The intermediate-amylose rice cultivar IM had a lower gelatinization temperature and faster gelatinization period than did the high-amylose rice cultivar DDS (Park et al., 2020). Under the same heat treatment, gelatinization properties may vary with rice cultivar amylose content and starch structural properties, such as short-chain ratio.

### 3.5. RS content of heat-treated brown rice flours

The RS content analyses of the four heat-treated BRF samples are listed in Table 2. The RS contents of RD, SBRD, SRRD, and SRRI were 13.0%, 12.1%, 14.0%, and 0.6%, respectively, and were significantly different from one another ( $P < 0.05$ ). In a previous study (Park et al., 2019), the intermediate-amylose IM and IP cultivars did not significantly differ in terms of RS content before and after roasting. Nevertheless, the RS content of DDS brown rice increased from 6–7%–13% after roasting at 240 °C for 10 min. In this study, heat treatment did not increase the RS content in SRRI but did increase the RS content in RD,

SBRD, and SRRD by ~12–14%. Among the DDS samples, the most highly gelatinized SBRD displayed the lowest RS content.

Roasting produces an agreeable aromatic flavor in rice and enables the grain to be consumed immediately. Food companies can use roasting to process foods economically, rapidly, conveniently, and simply (Ma, Boye, Azarnia, & Simpson, 2016). Both pH and moisture substantially affect RS (Kaur, Kaur, Sharma, & Jeet, 2018). One type of RS is produced by drying starch after it is cooked above its gelatinization temperature. High-moisture starch subjected to thermal gelatinization is prone to enzymatic hydrolysis. However, RS3 may be increased through starch retrogradation wherein the starch is cooled down to room temperature (25 °C) after gelatinization (García-Alonso, Jiménez-Escrig, Martín-Carrón, Bravo, & Saura-Calixto, 1999). Table 2 shows that the GD of SBRD increased more than that of SRRD, as the former was exposed to high moisture levels during the steam treatment. The RS content of SBRD decreased more than that of RD. However, the RS content was higher in all heat-treated DDS samples than in raw samples. The GD of SRRD was higher than that of RD as SRRD was retrograded by roasting after gelatinization. This process occurred primarily on the grain surface. Nonetheless, it had a positive impact on RS development. Amylose content might be the main factor influencing RS increase. A high-amylose raw material has a high RS content even after cooking (Meenu & Xu, 2019). However, the RS content of SRRI was <1% after heat treatment. Thus, the inherent structural properties of starch in the raw material cultivar are more important than the processing conditions required for RS formation.

### 3.6. Starch digestibility of heat-treated brown rice flours

The analyses of starch digestibility and the *in vitro* eGI for the four heat-treated BRF samples are illustrated in Fig. 4. RD and SRRD generated the lowest starch hydrolysis curves. Hence, their digestion was slow, and their digestibility was low. In contrast, SRRI and SBRD had high degrees of gelatinization and showed high starch hydrolysis curves, rapid starch digestion, and high starch digestibility rates. The eGI values of SRRI, SBRD, SRRD, and RD were 77.7, 62.8, 53.2, and 53.0, respectively, and were significantly different among each other ( $P < 0.05$ ).

Rapidly digestive starch (RDS) is digested within 20 min, slowly digestive starch (SDS) is digested within 20–120 min, and RS is not digested within 120 min (Englyst, Kingman, & Cummings, 1992). However, this classification does not accurately describe starch digestion. Determination of the glycemic index is an *in vivo* method for the measurement of postprandial blood sugar in response to the ingestion of foods containing carbohydrates (Jenkins et al., 1981) and helps evaluate the glycemic potential of functional foods. Overall, foods have low (<55), moderate (55–70), or high (>70) glycemic indices. Consumption of low-glycemic foods helps control type-2 diabetes mellitus (Tian & Sun, 2020). Moisture and heat gelatinize starch. The rate of starch hydrolysis may significantly increase after thermal processing. Fig. 4 demonstrates that the eGI values for RD and SRRD were lower than that for SBRD, whereas the latter had the highest GD. These findings are consistent with those of a previous study (Wang & Copeland, 2013). Samples from the same rice cultivar may have different degrees of digestibility depending on their processing methods. Hence, some of them could serve as low-glycemic functional foods.

Some studies have reported that the external factors affecting rice digestibility include storage, gelatinization, retrogradation, hydrothermal methods, such as heat-moisture treatment, and cooking methods, such as parboiling, whereas the intrinsic factors affecting rice digestibility include starch structure and protein and lipid contents (Toutounji et al., 2019). Syahariza, Sar, Hasjim, Tizzotti, and Gilbert (2013) claimed that amylose content is the most important factor affecting starch digestibility. Fig. 4a reveals that SRRD digestibility was significantly lower than that of SRRI, even though both samples were subjected to the same heat treatments. These results are consistent with those reported in an earlier study (Park et al., 2020), demonstrating that

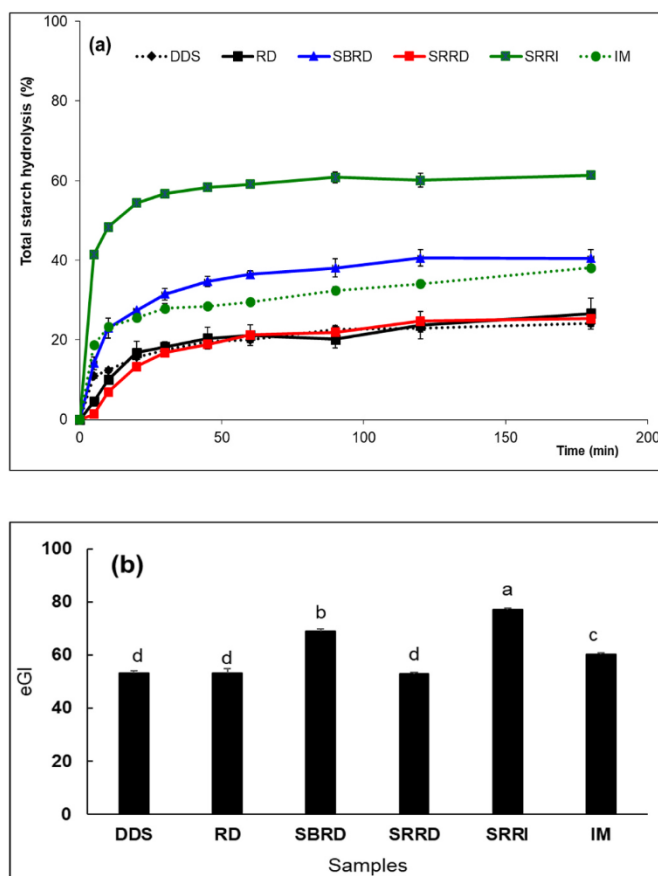


Fig. 4. Starch digestibility (a) and (b) estimated glycemic index (eGI) (b) of heat-treated brown rice flour (BRF). RD (roasted Dodamssal); SBRD (steamed brown rice and roasted Dodamssal); SRRD (steamed rough rice and roasted Dodamssal); SRRI (steamed rough rice and roasted Ilmi); DDS (untreated Dodamssal BRF); IM (untreated Ilmi BRF). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

a–d Values with different letters in a column are significantly different ( $P < 0.05$ ).

raw starch isolated from DDS had lower RDS but higher SDS and RS than did raw starch extracted from the intermediate-amylose IM cultivar. Zabidi and Aziz (2009) found that starch digestibility, HI, and eGI decrease with increasing RS content. The eGI values followed the same sequence as that of RS development shown in Table 2. The order of eGI was SRRD < RD < SBRD. Nevertheless, there was no significant difference between SRRD and RD. There was only 1% difference in RS content per sample. However, the eGI of SBRD was 9% higher than those of the others. Therefore, other important complex factors besides RS content influence starch digestibility. The SDS and RS fractions in roasted pea starch were significantly greater than those in the original raw material (Lu, Donner, & Liu, 2018). Steaming lowers RDS and raises SDS; however, these are strongly affected by GD (Toutounji et al., 2019). Fig. 4a shows that the elevated digestibility and RDS of high-GD SBRD might account for the high eGI of this material.

## 4. Conclusion

Brown rice was roasted after steaming rough rice to improve the quality of rice flour. Roasted brown rice treated after steaming DDS rough rice (SRRD) exhibited a greater RS formation than did roasted DDS brown rice (RD) or untreated DDS. Gelatinization was less in roasted (RD) than in steamed and roasted brown rice (all other treated samples, SRRD, SBRD, and SRRI), as the latter (RD) was exposed to



relatively less moisture. Moreover, the BRF derived from the former (SRRD) had the finest starch granules presenting with rough surfaces and less gelatinization than that derived from SBRD. Consequently, particle size diameters decreased during rice flour production. Hence, appropriate partial gelatinization, increased RS formation, and low eGI are important factors in the development of healthy functional foods from powdered rice. Herein, we confirmed that steaming rough rice and roasting brown rice (SRRD) resulted in BRF of superior quality compared with that derived from roasted brown rice (RD) or steamed and roasted brown rice (SBRD). The novelty of this study lies in its potential application in the production of powder-type simple meals using rice varieties with a C-type starch structure.

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## CRediT authorship contribution statement

**Jiyoung Park:** Conceptualization, Data curation, Writing – original draft, Project administration. **Sea-Kwan Oh:** Writing – review & editing, Supervision. **Hyun-Jung Chung:** Validation, Writing – review & editing. **Dong Sun Shin:** Software, Methodology. **Induck Choi:** Validation, Visualization. **Hyun-Jin Park:** Writing – review & editing, Supervision.

## Declaration of competing interest

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

## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.lwt.2022.113801>.

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