

## Rheological Properties of Different Degree of Pregelatinized Rice Flour Batter (Sifat Reologi bagi Larutan Bater Tepung Beras Tergelatin)

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### ABSTRACT

*The effect of using rice flour at different degree of gelatinization on rheological properties of a batter system was studied. Rice flour was gelatinized by heated for 20, 40, 60 and 90 min. The treated rice flour was used to prepare batters by stirring it for 2 min. The batter were subjected to determination of apparent viscosity, consistency and flow behavior index and storage modulus  $G'$ . The results showed that degree of gelatinization, DG of rice flour increased with the increased heating period up to 60 min. All rice flour batter shown shear thinning behavior since the viscosity decreased with increasing shear rate. The behavior was proven by the value of consistency coefficient,  $K$  more than 0 and flow behavior index,  $n$  less than 1. Rice flour batter heated in a longer period has higher viscosity and could be attributed by lower water content. The storage modulus,  $G'$  of heated rice flour were lower than  $G'$  of commercial and unheated rice flour. This result was probably because of the lower amylose and protein content in the modified starch pastes.*

**Keywords:** Degree of gelatinization; gelatinization; rheology; rice flour

### ABSTRAK

*Kesan menggunakan tepung beras pada darjah pengelatinan yang berbeza terhadap sifat reologi sistem bater telah dikaji. Tepung beras digelatinkan dengan memanaskannya selama 20, 40, 60 dan 90 min. Tepung beras yang terawat ini digunakan untuk menghasilkan larutan bater dengan mengacanya selama 2 min. Larutan bater ini digunakan untuk penentuan kelikatan, indeks konsistensi, indeks kelakuan aliran dan moduli keelastikan  $G'$ . Keputusan menunjukkan darjah pengelatinan, DG bagi tepung beras adalah meningkat apabila dipanaskan sehingga 60 min. Kesemua larutan bater tepung beras menunjukkan kelakuan penipisan ricihan disebabkan oleh kelikatannya menurun dengan peningkatan kadar ricihan. Kelakuan penipisan ricihan ini dibuktikan dengan nilai indeks konsistensi,  $K$  yang lebih daripada 0 dan indeks kelakuan aliran yang kurang daripada 1. Larutan bater beras yang dipanaskan lebih lama mempunyai kelikatan yang lebih tinggi dan mungkin disebabkan oleh kandungan lembapan yang kurang. Sifat kekenyalan,  $G'$  bagi tepung beras adalah lebih rendah daripada  $G'$  bagi tepung beras komersil dan tepung beras yang tidak dipanaskan. Penemuan ini mungkin disebabkan oleh kandungan amilosa dan protein yang rendah di dalam larutan kanji terubahsuai.*

**Kata kunci:** Darjah pengelatinan; pengelatinan; reologi; tepung beras

### INTRODUCTION

Gelatinization is a process when starch granules are heated in presence of liquid, resulting in the granules swelling and produces viscous fluids to impart the desired quality to food products. To obtain the desired quality of the products, an understanding of gelatinization process is a must. This process was studied by using differential scanning calorimetry (DSC) and have been reported by numerous researchers such as Miah et al. (2002); Nakorn et al. (2009); Ndife et al. (1998); Salma et al. (2011); Takahashi et al. (2005) and Vandeputte et al. (2003). Gelatinization process measured by using DSC was focused on gelatinization endothermic transition curve, degree of gelatinization, onset ( $T_o$ ), peak ( $T_p$ ) and end ( $T_e$ ) temperature in the endothermic curve.  $T_o$  is the temperature which rapid swelling starts in the starch granules.  $T_p$  is the temperature when the endothermic reaction occurs at the maximum, while the  $T_e$  is when all the starch granules

have fully gelatinized. Relationship between gelatinization process and microstructure of rice flour was established by scanning electron microscopic (SEM) images.

Few researchers have studied the rheological characteristic of rice flour batter system in order to determine the quality of the food product. Dogan et al. (2005) investigated the effects of soy and rice flour addition on batter rheology and quality of deep-fat fried chicken nuggets. The study was reported that coating pick-up was increased with increased of viscosity. The batter was found to be thixotropic behavior and reduced the oil absorption in fried chicken nugget. The addition of salt greatly affected the viscosity,  $G'$  and  $G''$  of batter system, as reported by Xue and Ngadi (2006). The increased of rice flour amount added into each batter system requires higher temperature and longer time to gelatinize starch. Dautant et al. (2007) cited rice flour with 21, 25 and 29% moisture (wet basis) and 3, 5 and 7% lipid content exhibited

a pseudoplastic flow behavior, as described by power law equation. Viturawong et al. (2008) studied effects of molecular weight of xanthan and salt on the rheological properties of rice starch. It was reported that all pastes exhibited time dependent shear thinning (thixotropic) with yield stress behavior decreased with the increased of molecular weight, but depended on salt added. The same result was also reported by Nakorn et al. (2009) that the apparent viscosity at 25°C for pregelatinized glutinous rice starch (PGS) and pregelatinized Jasmine rice starch (PJS) were decreased with increasing rate, as an indication of shear thinning behavior of the pastes. Lin et al. (2011) found the storage modulus,  $G'$  of rice flour was the highest compared to  $G'$  of rice starch and modified rice starch. As part of this literature studies, numerous studies have explored physicochemical properties of rice flour, but few studies have addressed the relationship between heating time of rice flour solution to the degree of gelatinization (DG) and these parameters. This study was carried out to determine the effect of heating time on gelatinization characteristics and rheological properties by varying the degree of gelatinization of the rice flour.

#### MATERIALS AND METHODS

The method of sample preparation is as by Baks et al. (2007) with some modifications. About 20 g of rice flour was mixed with 60 g of distilled water in order to get sample: water ratio at 1:3. The starch suspension was heated in 80°C water bath with overhead stirrer for 0, 20, 40, 60 and 90 min. The heated starch suspension was immediately immersed in the container with liquid nitrogen. After the sample was completely frozen, the sample was stored in a -30°C and freeze-dried in a freeze dryer until constant moisture content. The freeze-dried samples were grinded, sieved and stored in a desiccator before further use.

#### DETERMINATION OF DEGREE OF GELATINISATION

DSC measurement was performed using a Mettler-Toledo DSC 822 instrument according to Salma et al. (2011) with some modifications. Freeze-dried starch samples were weighed in a aluminum DSC pan and distilled water was added to obtain a ratio of 1:3 (sample:water) starch pastes. The pan was hermetically sealed and was stabilized for 1 h. The pan was then heated in the range of 25 to 120°C at a scanning rate of 10°C min<sup>-1</sup>. An empty pan was used as a reference. For each measurement, normalized enthalpy,  $T_o$ ,  $T_p$  and  $T_e$  were determined. The data obtained from the analysis were evaluated by using STAR software. The DG was calculated using the following formula:

$$\text{Degree of Gelatinization, DG} = 100 - (\Delta H_t / \Delta H_0 \times 100),$$

where  $\Delta H_t$  is the normalized gelatinization enthalpy after  $t$  minutes of heating while  $\Delta H_0$  is the normalized gelatinization enthalpy without heating.

Commerical rice flour, unheated and heated rice flour microstructures were observed using a scanning electron microscope (Topcon SM-300) at 8kV accelerating voltage with sputter coating. The working specimen working distance was 9 mm. Secondary electron images were recorded digitally at a magnification of 2000x.

Flow and viscoelastic behaviours of the batter were performed by using a rheometer (Physica MCR 301, Anton Paar) with a cone and plate geometry sensor (1 cone angle, 500 mm diameter and 0.05 m gap). The sample was placed in the rheometer at 25°C.

#### FLOW BEHAVIOR

Apparent viscosity was measured as a function of shear rate over the range 0.3 to 300s<sup>-1</sup>. The result was reported as the mean of three replicates on each sample.

#### VISCOELASTIC BEHAVIOR

Changes due to the effect of cooking temperature on the batters, the storage modulus ( $G'$ ) and loss modulus ( $G''$ ) were monitored as functions of temperature and determined using dynamic oscillatory test. After equilibration at the initial temperature of 25°C for 5 min, the samples were heated continuously at 2.5°C/min to a final temperature of 100°C. During the heating process, the samples were sheared at a fixed frequency of 1 Hz and strain amplitude of 1%. The storage modulus ( $G'$ ) and the loss modulus ( $G''$ ) were continuously monitored during the dynamic rheological testing. Data related to dynamic viscoelastic characteristics of samples during gelatinization were gelatinization temperature ( $T_g$ ) and peak storage modulus ( $G'_p$ ). Test was repeated at least three times.

All the analysis were done and reported by mean values. Data were assessed by analysis of variance (ANOVA) and Duncan's multiple comparison test with the level of significance ( $p < 0.05$ ) were used to determine the differences between the effects of heating period on quality parameters of the samples.

#### RESULTS AND DISCUSSION

Figure 1 shows the degree of gelatinized (DG) of rice flour after 0, 20, 40, 60 and 90 min of heating. Rice flour without heating treatment was calculated as zero DG. DG for all treatment of samples increased with the increasing heating time up to 60 min. After 60 min of heating, rice flour did not show significant difference in DG. The highest DG obtained from 90 min heating period was 83.74% indicating that 16.26% of the starch remained ungelatinized after 90 min of heating. Overall, the results shown in this study were consistent with those reported by Miah et al. (2002), Ndife et al. (1998) and Salma et al. (2011) where increased heating time was found to increase DG. The result from Figure 1 was also consistent with the result shown in Figure 2. In Figure 2, there were reductions in the endotherm peak surfaces from rice flour without heating treatment to the

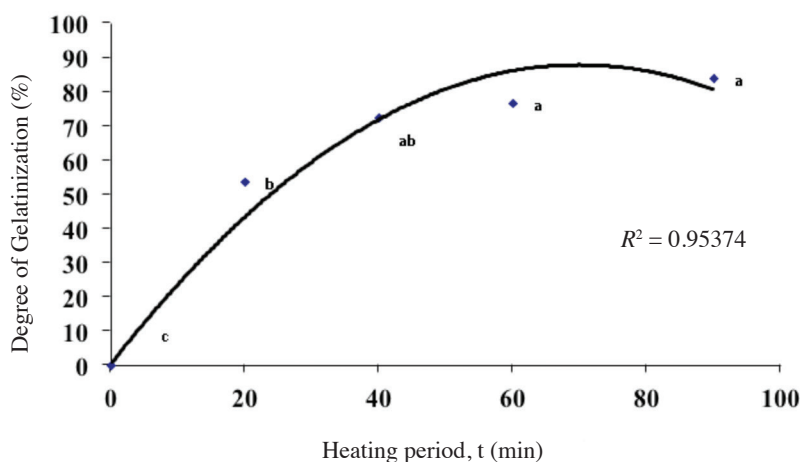


FIGURE 1. Degree of gelatinization of different heating time of pregelatinized rice flour samples. Mean with different letter are significantly different ( $p < 0.05$ )

rice flour heated for 90 min. Miah et al. (2002) presumed that the early part of gelatinization is the uptake of water by the amorphous background region and rapid expansion of the starch granules. This is followed by disruption of granules structures (Aini & Purwiyatno 2010). Unheated sample (0 min) was not gelatinized and it could be seen that size of the peak was the highest among the treatments. Increased heating time allowed more starch granules to swell and became ruptured. At the 60 and 90 min heating time, the samples were nearly fully gelatinized as shown by the lacking of gelatinization endotherm in Figure 2 when heating time reached 60 and 90 min. The decreased of the endotherm peak size from unheated sample to 90 min heated sample were also consistent to the result in Table 1. From Table 1, the gelatinization temperatures were higher as heating time increased.  $T_o$  of samples ranged from 72.0 to 90.6°C.  $T_p$  of samples ranged from 77.0 to 96.4°C while  $T_e$  of samples ranged from 81.0 to 103.9°C. From statistical analysis it was found that  $T_p$  and  $T_e$  of samples heated for 0 and 20 min were significantly different with samples heated for 40, 60 and 90 min. Above 40 min of heating, there was no significant difference in  $T_p$  and  $T_e$ . This result reveals that all samples were consistent. While,  $T_o$  of unheated rice flour sample was significantly different with heated rice flour sample.  $T_o$  of 20 min heating time sample did not show significant different with sample heated for 40 min but was significantly different to the sample heated 60 min and above. This partial gelatinization allowed the diffused amylose molecules to reorient and surround the amylopectin granules (which were already swollen by the penetration of water molecules) and subsequently retrograded on cooling and storage (Miah et al. 2002). The retrogradation provides structural stability and makes starch gelatinization difficult to occur resulting in higher gelatinization transition temperature (Barichello et al. 1990). Despite of all results mentioned above, Figure 1 also shows there were some gelatinization process occurred at the temperature below 60°C for all samples. This was probably because of some weak granules produced when

the rice flour were heated. From Figure 1, it could be seen that increased heating period also increased the peak size for these weak granules. Apparently, heating treatment weakened the starch granules.

Figure 3(a) to 3(f) shows SEM images of microstructure of rice flours. From the image, the morphological structure of commercial rice flour (Figure 3(a)) was round and spherical of 1.8 to 5.6  $\mu\text{m}$  in size. The small granules were clustered to form granule populations. The result was similar as reported by Nakorn et al. (2009) and Takahashi et al. (2005). Meanwhile, the morphological structure of rice flour without heating treatment (Figure 3(b)) looked similar with commercial rice flour but bigger in size (approximately 3.9 to 6.4  $\mu\text{m}$ ). The differences in granule size between these two flours were caused by treatment applied to the rice flour without heating period. This rice flour was mixed with water prior freeze dried caused the expansion in granule size. The heating treatment affected the structural changes to the other rice flour samples. Figure 3(c) to Figure 3(f) shows the structural changes in 20, 40, 60 and 90 min heating treatments. The morphological structures were spherical with some loss of granular characteristics when rice flour was heated in 20 min. It is presumed that the starch granules absorbed water, swelled and ruptured at different rates. The sizes of the diameter were approximately 4.7 to 7.4  $\mu\text{m}$ . On the other hand, most of the granules were ruptured when rice flour was heated in 40 min. The diameter sizes ranged from 3.2 to 8.5  $\mu\text{m}$ . Finally, the rice flour granules were totally disintegrated when heated in 60 and 90 min indicating completed gelatinization of starch. The release of soluble component (Nakorn et al. 2009) and amylose during thermal treatment were resulted in hollowness of the starch granules which may be responsible for better hydration of processed starches (Yadav et al. 2006).

Flow behavior of the rice flour batters were examined by measuring the viscosity shear stress with shear rate. For the range of the shear rate used in this study, it could be seen in Figure 4 that batter viscosity decreased with

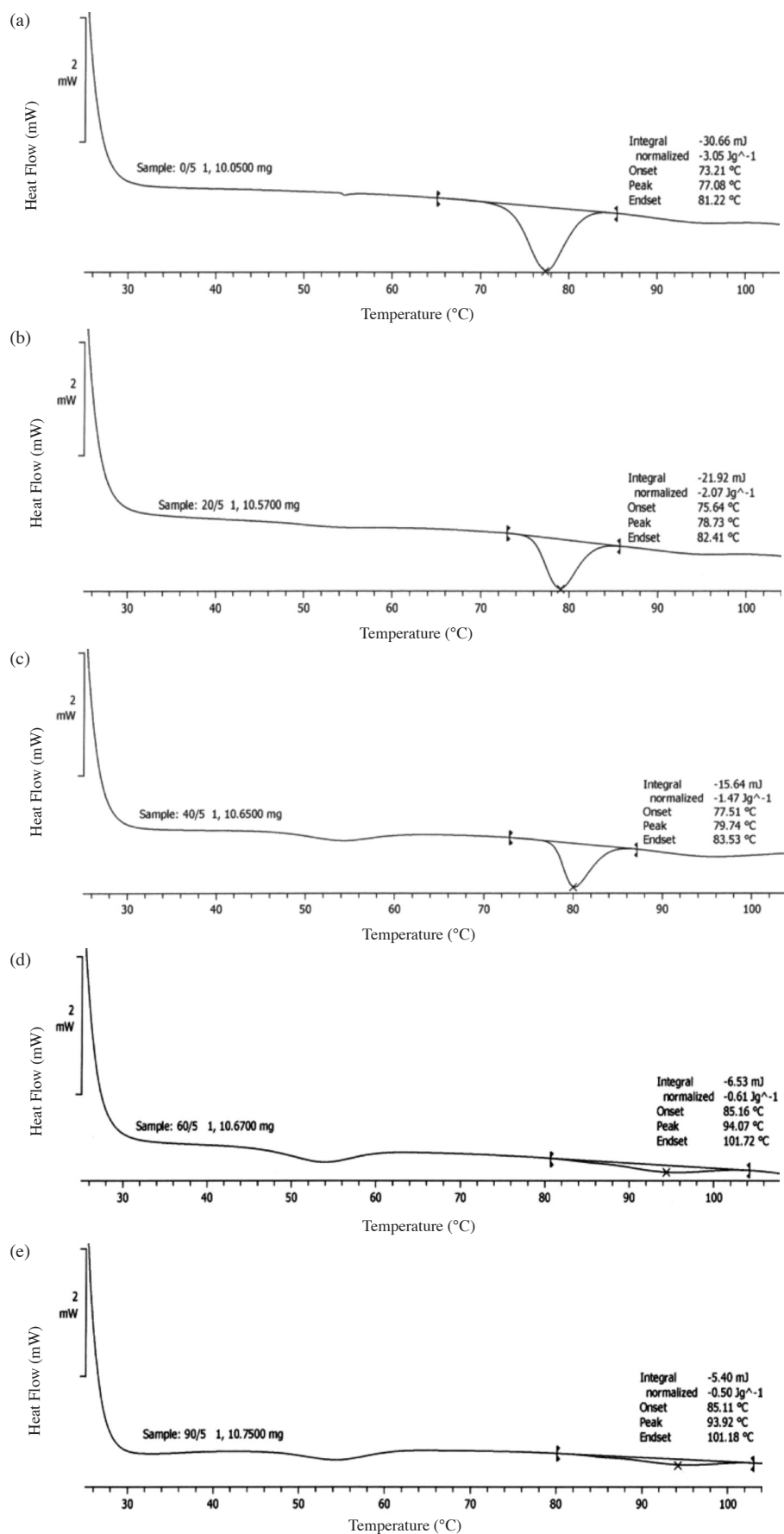


FIGURE 2. DSC endotherms of (a) unheated rice flour (starch-water ratio 1:3 w/w) and pregelatinised rice flour heated in 80°C water bath for (b) 20; (c) 40; (d) 60 and (e) 90 min



TABLE 1. Effect heating time on onset ( $T_o$ ), peak ( $T_p$ ) and end ( $T_e$ ) temperature

Heating Time	$T_o$ (°C)	$T_p$ (°C)	$T_e$ (°C)
0	73.2 <sup>c</sup>	77.1 <sup>b</sup>	81.6 <sup>b</sup>
20	79.1 <sup>b</sup>	82.3 <sup>b</sup>	87.0 <sup>b</sup>
40	82.8 <sup>ab</sup>	89.1 <sup>a</sup>	95.2 <sup>a</sup>
60	85.7 <sup>a</sup>	92.0 <sup>a</sup>	97.8 <sup>a</sup>
90	85.8 <sup>a</sup>	94.7 <sup>a</sup>	101.6 <sup>a</sup>

Mean with different letter are significantly different ( $p < 0.05$ )

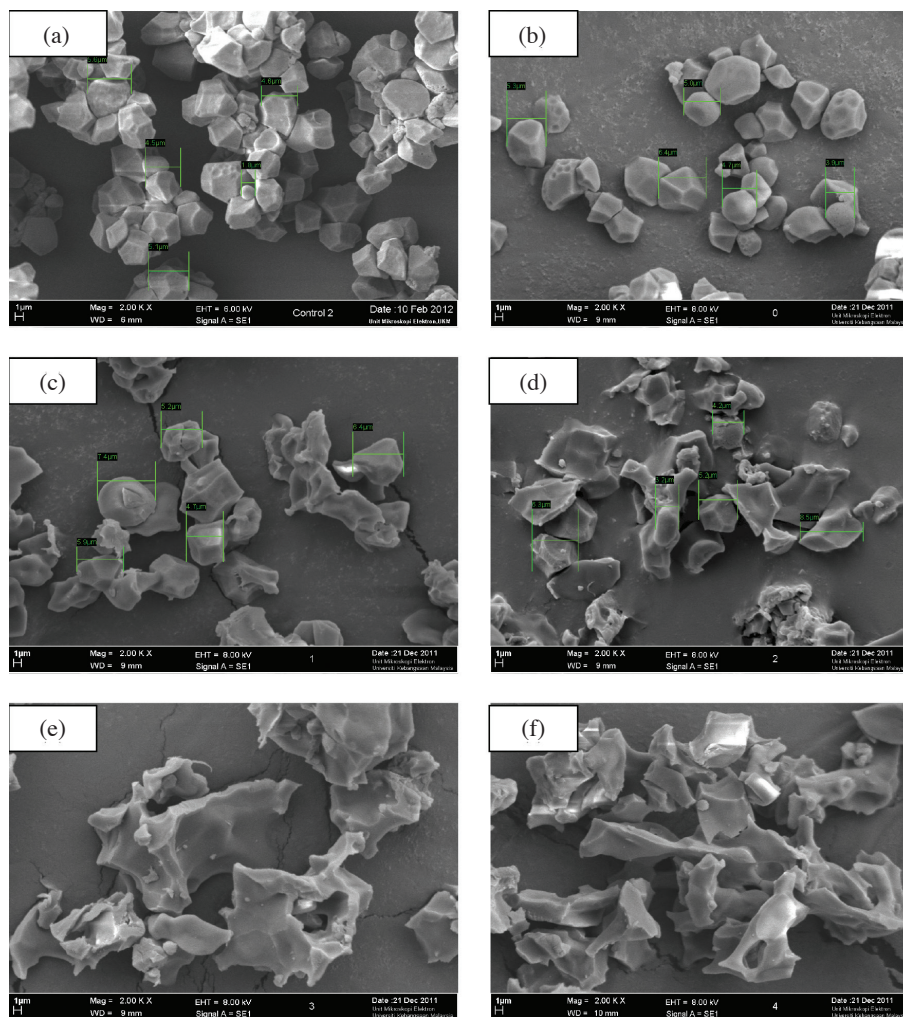


FIGURE 3. Scanning electron microscopy images of rice flours: (a) commercial rice flour (Erawan Brand); and rice flour with (b) 0; (c) 20; (d) 40; (e) 60 and (f) 90 min heating period

increasing shear rate, revealing the shear thinning behavior of the fluid. This result was similar as reported by Dogan et al. (2005) and Xue and Ngadi (2006) that the rice flour batter was found to be non-newtonian and could be modeled as power law fluid. The increased shear rate deforms and rearranges starch particles, resulting in lower flow resistance and consequently lower viscosity. Shear thinning behavior is very common in fruit and vegetable product and could be modeled as a power law fluid. Figure

5 shows all batter have thixotropic behavior since viscosity of batters reduced with increased over time. This result was similar to the result reported by Dogan et al. (2005). Figures 4 and 5 also reveal that rice flour batter heated in a longer period has higher viscosity. The rice flour heated in 90 min showed highest viscosity. This could be attributed to the released of soluble component and amylose and thus decreased free water in the batter system. Apparently, a higher viscosity is attributed by lower water content.

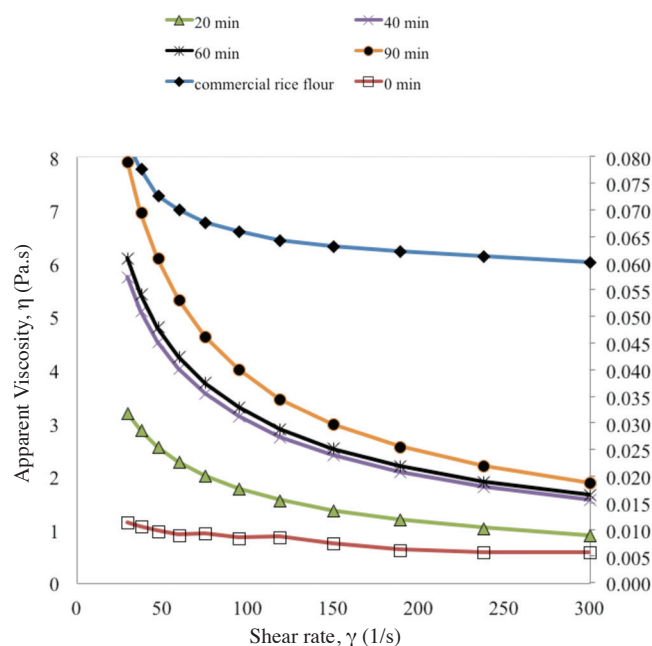


FIGURE 4. Change in rice flour batter viscosity with shear rate when different heating period were applied to the rice flour slurries

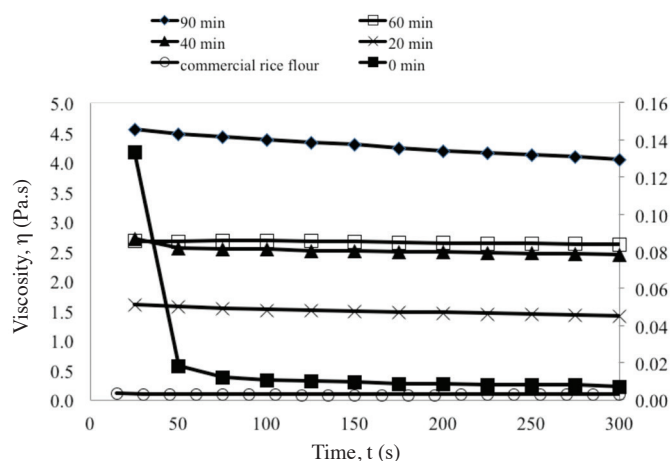


FIGURE 5. Change in rice flour batter viscosity with time when different heating period were applied to the rice flour slurries

Furthermore, the viscosity of rice flour heated at 60 and 90 min were significantly different with rice flour batter heated in 20 min, indicating that rice flour particles heated in 20 min do not hydrated as fully and do not ruptured as much as rice flour heated in 60 and 90 min. This result was proven by SEM images in Figure 3. Viscosity of rice flour with heat treatment also shows significantly different from unheated rice flour and commercial rice flour. This result indicated that heat treatment caused starch granules swell, absorbed more water thus decreased the free available water in the system and developed more apparent viscosity than unheated rice flour. Kuenchan et al. (2009) reported that starch that was disrupted was exhibited higher swelling and developed more apparent viscosity than native starch.

As mentioned above, the rheological behavior of all rice flours follow a power law model, the effect of heating period on the consistency coefficient,  $K$  and flow behavior index,  $n$  were also studied. From Table 2, it can be stated that consistency coefficient were more than zero and flow behavior index were less than 1, confirmed the shear thinning behavior of the rice flour batter. This result was similar as reported by Dogan et al. (2005). The increased heating period significantly increased consistency coefficient. The increased consistency coefficient were related to the increased viscosity (Chen et al. 2008) and also increased amount of force needed for the rice batter to flow (Holdsworth 1996). It means that the increased viscosity and force need for the rice flour batter to flow will result

TABLE 2. Average viscosity, consistency coefficient, K and flow behavior index, n of rice flour batters at different heating period. Mean with different letter are significantly different ( $p < 0.05$ )

Rice flour	Average viscosity	Consistency coefficient, K (Pa.s <sup>n</sup> )	Flow behaviour index, n
Commercial rice flour	0.0681 <sup>c</sup>	0.12 <sup>d</sup>	0.8867 <sup>a</sup>
0 min	0.0083 <sup>c</sup>	0.06 <sup>d</sup>	0.5845 <sup>b</sup>
20 min	1.8766 <sup>b</sup>	18.06 <sup>cd</sup>	0.6607 <sup>a</sup>
40 min	3.3376 <sup>bc</sup>	40.85 <sup>cb</sup>	0.4432 <sup>b</sup>
60 min	3.5309 <sup>a</sup>	43.94 <sup>b</sup>	0.4386 <sup>b</sup>
90 min	4.3630 <sup>a</sup>	67.95 <sup>a</sup>	0.3762 <sup>b</sup>

in enhanced batter pick up. Inspection of the flow behavior index, n showed that flow behavior index did not exhibit a significant different with the ranged heating period and also to the commercial rice flour. It is presumed that flow behavior index may be considered a time independent parameter as well as a temperature independent parameter as reported by Dautant et al. (2007).

Based on Figure 6, there were three level of storage modulus,  $G'$  when commercial and unheated rice flour were heated from 25 to 100°C. At the temperature below than 73.2°C, there was a constant  $G'$  of commercial rice flour at 1.3 kPa and then was an increased when rice flour was heated to 73.2°C. At the temperature of 77.7°C,  $G'$  of commercial rice flour reached a maximum  $G'$  (40 kPa) and then decreased. For the unheated rice flour, the  $G'$  was 200 Pa until it was heated to 75°C. After 75°C, the sample  $G'$  was drastically increased to and reached a maximum  $G'$  at 3.28 kPa. Peak temperature of this  $G'$  was 80.4°C. The  $G'$  of the sample was gradually increased and became constant at 3.71 kPa. At temperature of 85°C,  $G'$  of unheated rice

flour decreased. Chen et al. (2008) classified the first stage of  $G'$  change as the level of the starch molecules absorbed energy resulting in rice flour batter softer. The  $G'$  characteristics at the second level was caused by gluten denaturation and starch swelling. The third level of  $G'$  curve pattern was resulted by mixture stability caused by gelatinized protein and starch molecules. It can be stated that the level of starch granules swelling leads to the gelatinization process.

On the other hand, the  $G'$  pattern for rice flour heated in 20 min is quite different with the commercial and unheated rice flour. The  $G'$  of 20 min heated rice flour was gradually increased until 77°C and then drastically increased until 84.5°C. The  $G'$  of the rice flour was being constant at maximum 4.8 kPa when the batter was heated to 86°C and finally decreased. Trend  $G'$  for rice flour samples heated in 60 and 90 min were almost similar to each other. This result was caused by full gelatinization of starch granules and created a stable starch paste. From Figure 6 it could be seen that  $G'$  of heated rice flour were lower than  $G'$  of commercial

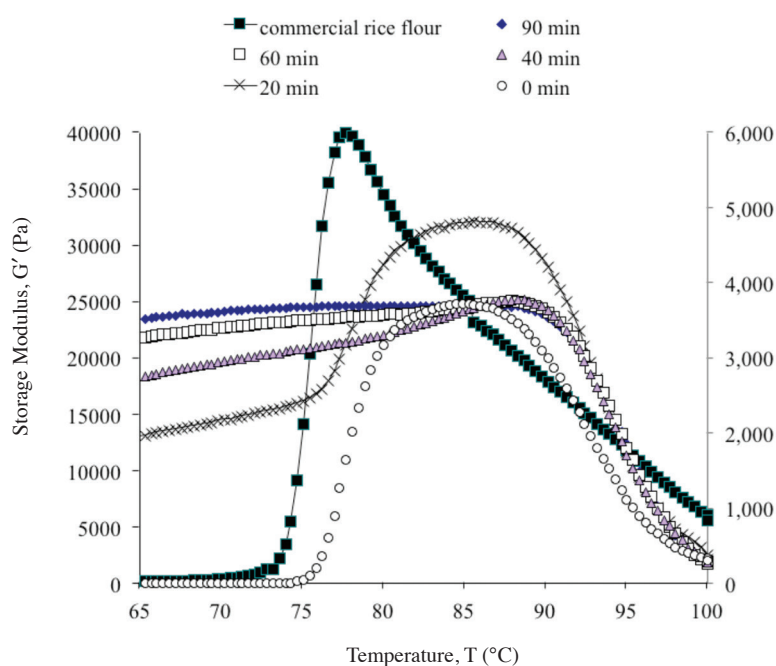


FIGURE 6. Change in rice flour batter storage modulus with temperature when different heating period were applied to the rice flour batter

and unheated rice flour. This result was probably because of the great amount of amylose leached out by heat treatment lowered the  $G'$  in heated rice flours. Lin et al. (2011) relate the lower  $G'$  in modified starch to the lower protein content, whereas, the protein in the starch enhanced the formation of the gel network resulting in increased the hardness and strength of the starch paste.

### CONCLUSION

This study investigated the effect of heating time on gelatinization characteristics and rheological properties by varying the degree of gelatinization of the rice flour. It was found that degree of gelatinization, DG of rice flour increased with the increased heating period up to 60 min. After 60 min of heating, rice flour did not show significant difference in DG. The endotherm peak surfaces reduced from rice flour without heating treatment to the rice flour heated for 90 min. The gelatinization temperatures ( $T_o$ ,  $T_p$  and  $T_e$ ) were higher with the longer heating period. The starch granules also became swelled, disintegrated and lost the spherical form when heated in longer period, indicating the increased degree of gelatinization of the rice flour. All rice flour batter shown shear thinning behavior since the viscosity decreased with increasing shear rate. The behavior was proven by the value of consistency coefficient,  $K$  more than 0 and flow behavior index,  $n$  less than 1. Rice flour batter heated in a longer period has higher viscosity and could be attributed by lower water content. The storage modulus,  $G'$  of heated rice flour were lower than  $G'$  of commercial and unheated rice flour. This result was probably because of the lower amylose and protein content in the modified starch pastes.

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