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# Rheological properties of pastes and gels of rice flour with varied amylose contents

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#### **ABSTRACT**

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Udomrati, S., Tungtrakul, P., Lowithun, N., and Thirathumthavorn, D. (2022). Rheological properties of pastes and gels of rice flour with varied amylose contents. Science, Engineering and Health Studies, 16, 22030007. The rheological behaviors of gelatinized pastes and gels, respectively, prepared from 5% w/w and 30% w/w rice flour with varied amylose contents and obtained from different rice varieties, were monitored using steady shear and dynamic shear rheological determinations. All of the gelatinized pastes (5% w/w) exhibited a shearthinning behavior and a less solid-like behavior (storage modulus (G') <100 Pa). RD41, which had a high amylose content, had the lowest viscosity and G'values at low flour concentration (5% w/w due to low amylose leaching and lack of gel formation. When its concentration was increaseds to (30% w/w, RD41 formed the firmest gel, compared with the rice varieties with low (PT1 and RD43) and intermediate (KTH17) amylose contents. The G' value for all samples produced from 30% w/w flour was considerably higher (200-4,000 times) than that of the system produced using 5% w/w, indicating gel formation; the exception is RD6 (glutinous), which remained to be a flowable paste. The results showed a positive linear correlation between G' and amylose content. Rice variety, amylose content, and flour concentration were the factors affecting the rheological behaviour of rice flours.

Keywords: amylose; rice flour; rheological properties; paste; gel

## 1. INTRODUCTION

Rice is consumed as a staple grain in many Asian countries, and rice flour serves as the main ingredient in various food products. Rice is a source of carbohydrate, and it contains non-allergic protein, high amount of unsaturated fatty acids (>70%) and bioactive compounds, such as  $\lambda$ -oryzanol, vitamin E and phytosterols (Naivikul and Tungtrakul, 2014). Rice flour is widely used in food and nonfood industries given its low price, large-scale production, and acceptance by customers (Naivikul and Tungtrakul, 2014). Today, customers strongly prefer healthy food

products that are made from natural ingredients and that are only mildly processed. Thus, native flours (e.g., rice flour) have been increasingly becoming attractive in the food industry and have been considered an important component in various food products (Techawipharat et al., 2008).

When flour is heated in excessive amounts of water, its starch granules swell and the crystalline structure disintegrates because of starch gelatinization, forming a viscoelastic mass. As the system cools, starch retrogradation occurs. The amylose molecules undergo inter- and intramolecular interactions to form a three-dimensional network



within a short period; during long-term storage, amylopectin re-crystallizes nearly entirely via intermolecular interactions (Putaux et al., 2000).

The viscoelastic behavior of a system during gelatinization and retrogradation produces marked changes that can be detected and monitored using rheological approaches (Li and Zhu, 2018). Small oscillatory frequency and temperature sweep experiments are simple deformation rheological tests performed within the linear viscoelasticity region (LVR), and they monitor quite rapid molecular motions that do not involve specific intermolecular interactions (Doublier et al., 1992), providing sensitive microstructural information about samples subjected to different temperatures and frequencies (Ye et al., 2016). The rheological properties of rice flour are influenced by various factors, such as rice variety, amylose content, the structure of starch molecules (Kowittaya and Lumdubwong, 2014), particle size and particle size distribution (Ahmed et al., 2015), and flour concentration (Ahmed et al., 2008). Rheological information may be used to evaluate gelation behavior, correlate textural attributes to the design of new food products, analyze flow conditions and process design, control the quality of final products, and predict product stability (Li et al., 2014; Xie et al., 2008; Liu et al., 2015; Mandala et al., 2004). The selection of suitable ingredients significantly impacts the design and control of the textural characteristics of various food products and consequently enables them to achieve their desired functionality and unique properties. The rheological properties of food ingredients during heating and cooling provide important information for the handling, designing and operation of food processing equipment (e.g., mixing and piping equipment and pumps). Moreover, rheological data from oscillatory frequency sweep tests are used to estimate the stability of food products. Understanding the rheological properties of rice flour is important, and knowledge of such properties is very useful. Thus, through steady shear and dynamic shear rheological determinations, this research aimed to investigate the rheological behavior of rice flours obtained from various rice varieties with different amylose contents and used at varying concentrations.

# 2. MATERIALS AND METHODS

## 2.1 Materials

Five Thai rice varieties with different amylose contents were investigated: Kao Khor 6 (RD6) is a glutinous rice variety; Pathum Thani 1 (PT1) and Kao Khor 43 (RD43) are varieties with low amylose contents; Khao Tah Haeng 17 (KTH17) has an intermediate amylose content; and Kao Khor 41 (RD41) exhibits high amylose content. All of these rice varieties were cultivated in 2017. The experiment was performed in 2017-2018.

## 2.2 Rice flour preparation

Rice grains were soaked in water at room temperature ( $\sim 30$ °C) for 3 h. The soaked grains were milled using a stone miller at a rice grain-to-water ratio of 1:2. The obtained rice slurry was centrifuged to obtain the rice cake, which was dried in a hot air oven overnight at 40°C. The dried rice flour was ground using a mortar and then passed through a 100-mesh sifter. The sieved flour samples were

kept in sealed polypropylene bags and stored at  $4^{\circ}\text{C}$  before further analysis.

## 2.3 Amylose content

An amylose/amylopectin assay kit (MegaZyme Pty Ltd., New South Wales, Australia) was used to determine the flours' amylose content.

## 2.4 Proximate analysis of rice flours

The moisture, protein, and fat contents of the rice flour samples were determined using the methods described by the AACC (2000).

# 2.5 Gelatinization determination using differential scanning calorimetry (DSC)

The gelatinization transition enthalpy ( $\Delta H$ , expressed in J/g) and the melting temperature in gelatinization (onset temperature ( $T_o$ ), peak temperature ( $T_p$ ), and conclusion temperature ( $T_c$ )) were determined using a DSC equipment (Diamond DSC, Perkin-Elmer, California, USA). The rice flour samples (2-2.5 mg) were weighed into the DSC equipment pan, then distilled water was added (the flour-to-water ratio was 30:70 (dry basis)). The DSC pan containing the sample was sealed and heated from 25°C to 95°C at  $10^{\circ}$ C/min.

## 2.6 Rheological properties

All rheological property determinations for the rice flour samples were performed with a rheometer (MCR 305, Anton-Paar, Graz, Austria) using a parallel plate geometry with a diameter of 50 mm and a gap distance of 1 mm. Each sample of approximately 2 mL was transferred to the bottom plate of the rheometer, and the edge of the sample was covered with paraffin oil to prevent dehydration during the experiments. The sample temperature was controlled using a Peltier system, which has an accuracy of  $\pm 0.1^{\circ}\text{C}$ .

# 2.6.1 Rice flour system with a low flour concentration (paste system)

A rice flour suspension (5% w/w (dry basis)) was capped and heated in boiling water for 35 min until fully gelatinized. The sample was gently shaken to prevent rice granule precipitation. Then, the gelatinized rice paste was placed in a water bath at room temperature for 30 min. Rheological determination was performed with two replications. Measurements were made in each replication, and data were collected three times.

## 2.6.1.1 Steady shear properties

The viscosity of each gelatinized rice paste sample as a function of shear rate was determined at 25°C. The shear rate ranged from  $0.01~\rm s^{-1}$  to  $100~\rm s^{-1}$ . Experimental data were fitted to the Herschel-Bulkley model as follows:

$$\tau = \tau_0 + k \cdot \gamma^n \tag{1}$$

where  $\tau$  is the shear stress (Pa),  $\tau_0$  is the yield stress (Pa),  $\gamma$  is the shear rate (s<sup>-1</sup>), n is the dimensionless flow behavior index, and k is the consistency index (Pa.s<sup>n</sup>).

## 2.6.1.2 Oscillatory strain sweep test

The experiment was performed at a constant frequency of 1 Hz with a strain sweep of 0.001-10% at  $25^{\circ}$ C. The storage or elastic modulus (G', Pa) and loss or viscous modulus (G'',



Pa) were monitored. The limit of the LVR was determined in order to carry out the frequency sweep test.

#### 2.6.1.3 Oscillatory frequency sweep test

The oscillation test was performed within the LVR for all samples. A frequency sweep of 0.01-10 Hz was conducted at 25°C with a constant strain of 0.1%. G', G'', loss tangent (tan  $\delta = G''/G'$ ), and complex viscosity ( $\eta^*$ ) values were automatically determined as a function of frequency. The  $\eta^*$  value was calculated by the following correlation (Bird et al., 1987):

$$\eta^* = \left[ \left( \frac{G'}{\omega} \right)^2 + \left( \frac{G''}{\omega} \right)^2 \right]^{\frac{1}{2}} \tag{2}$$

where  $\omega$  is oscillation frequency in radians.

# 2.6.2 Rice flour system with a high flour concentration (gel system)

Rice flour suspensions were prepared using 30% w/w (dry basis) flour concentration and stirred using a vortex mixer at room temperature for 5 min. Subsequently, the flour suspension (approximately 2 mL) was loaded onto the bottom plate of the rheometer.

# 2.6.2.1 Oscillatory temperature sweep test and oscillatory frequency sweep test

Each rice flour suspension sample was initially equilibrated at 25°C for 5 min and then heated at 25-95°C at a rate of 5°C/min, and a frequency of 1 Hz and strain of 0.1% were applied. The G', G'', tan  $\delta$ , and  $\eta^*$  values as function of temperature were determined. Then, the gelatinized sample was cooled to 25°C for further frequency sweep testing. A frequency sweep of 0.1-10 Hz was applied with a constant strain of 0.1% at 25°C. The G', G'', tan  $\delta$  and  $\eta^*$  values as a function of frequency were determined.

# **Table 1.** Composition of different varieties of rice flour

# 2.6.2.2 Oscillatory frequency sweep test at different heating temperatures

The rice flour suspension (30% w/w) was heated up to 60°C, 70°C and 80°C and equilibrated for 5 min. A frequency sweep of 0.1-10 Hz was performed with a constant strain of 0.1% at fixed temperatures (60°C, 70°C and 80°C). The G', G'', tan  $\delta$ , and  $\eta^*$  values as function of frequency were determined.

#### 2.7 Statistical analysis

Data were subjected to analysis of variance (ANOVA) and Duncan's multiple range test at the 95% confidence level (p<0.05) using the SPSS statistical software (IBM Corp., New York, USA).

# 3. RESULTS AND DISCUSSION

# **3.1** Composition of rice flours prepared from various rice varieties

The amylose contents of all rice flours are shown in Table 1. The five rice varieties could be categorized into four groups based on their amylose contents. RD6, which is a glutinous rice variety, contained 3% amylose. PT1 and RD43 had low amylose contents at 14% and 16%, respectively. KTH17 exhibited intermediate amylose content at 21%, and RD41 had a high amylose content at 30% (Juliano, 1992). The rice flours obtained from these five varieties had moisture, fat, and protein contents of 11-12%, 0.3-0.8% and 7-11% (dry basis), respectively, as shown in Table 1. The suitable moisture content of rice flour in order to inhibit microbial growth during storage is below 13% (TISI, 2020). Meanwhile, RD41 had the highest protein contents, and RD6 and RD43 had the lowest protein content. PT1 had the lowest fat content, and RD43 had the highest fat content.

Rice variety	Amylose (% dry basis)	Moisture (% wet basis)	Fat (% dry basis)	Protein (% dry basis)
RD6	$3.68^{d} \pm 0.63$	11.42 ± 0.69	$0.44^{bc} \pm 0.18$	7.40° ± 0.53
PT1	14.46° ± 0.01	11.47 ± 0.28	$0.37^{c} \pm 0.02$	$8.47^{\rm b} \pm 0.15$
RD43	15.25° ± 0.41	11.31 ± 0.62	$0.77^a \pm 0.03$	$7.70^{\circ} \pm 0.00$
KTH17	$19.81^{\rm b} \pm 0.01$	11.59 ± 0.67	$0.41^{c} \pm 0.02$	$10.46^{a} \pm 0.05$
RD41	$29.89^{a} \pm 0.07$	11.45 ± 0.18	$0.61^{ab} \pm 0.03$	11.10a ± 0.22

Note: \*Different letters (a,b,c,..) within a same column indicate significant difference at the 95% confidence level. Means ± standard deviation were shown.

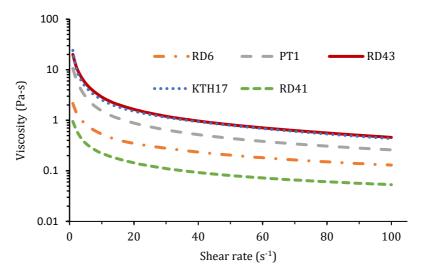
# 3.2 Rheological properties of gelatinized rice flours

# 3.2.1 Low rice flour concentration (paste system)

At 5% w/w rice flour concentration, the rice samples formed a flowable gelatinized paste; specifically, PT1, RD43, and KTH17 had a high viscosity, RD6 had an intermediate viscosity, and RD41 had a low viscosity, as shown in Figure 1. The k values of the rice flour samples are shown in Table 2. The k value was strongly positively related to viscosity because k is an indicator of the viscosity

of fluids. During gelatinization, the leaching of starch molecules from gelatinized starch granules and the swelling of granules are the main factors that increase the viscosity of a system, as these factors impart flow resistance. None of the completely gelatinized samples were able to set as a gel, possibly because the flour concentration (5% w/w) in the diluted system was too low; hence, there were insufficient starch molecules that would interact and become entangled to form a gel network.





**Figure 1.** Flow curves of different varieties of gelatinized rice paste (5% w/w flour)

Table 2. Rheological characteristics of gelatinized flour pastes (5% w/w) from different rice varieties

Rice variety	Flow behavior index (n)	Consistency index (k) (Pa.s <sup>n</sup> )
RD6	$0.387^{a} \pm 0.022$	4.41° ± 1.56
PT1	$0.125^{\circ} \pm 0.015$	23.27b ± 6.34
RD43	$0.139^{\circ} \pm 0.036$	33.63 <sup>a</sup> ± 10.31
KTH17	$0.153^{\circ} \pm 0.007$	$27.46^{ab} \pm 0.08$
RD41	$0.340^{\rm b} \pm 0.014$	$1.19^{c} \pm 0.14$

Note: \*Different letters (a,b,c,..) within a same column indicate significantly different at the 95% confidence level. Means ± standard deviation were shown.

The viscosity of the completely gelatinized rice paste of all the rice varieties decreased as the shear rate increased (Figure 1); thus, the pastes exhibited a shearthinning behavior. The shear-thinning behavior was confirmed by the *n*, which was less than 1 for all samples, as shown in Table 2. This result agreed with that obtained by Prasad et al. (2013), who reported that rice flour (0.5-10% w/w) exhibited a shear-thinning behavior with an nrange of 0.46-0.76. A shear-thinning behavior indicates the deformation of starch entanglement or the disruption of weak-swollen gelatinized starch granules or both. Starch molecules are elongated, and starch granule fragments are aligned in a high shear field, resulting in reduced viscosity because of the reduced volume fraction of starch agglomeration and swollen granules (Lin et al., 2010). The n values for RD6 and RD41 were higher (indicating a weaker shear-thinning behavior) than those for the other samples (PT1, RD43, and KTH17), possibly because of the considerably lower amylose content in RD6 (glutinous rice); consequently, the amount of leached amylose was considerably lower (or even none) to induce starch entanglement. However, the gelatinized paste of RD41 (with a high amylose content at ~30%) might have contained a small amount of leached starch polymers (amylose and amylopectin), resulting in less starch entanglement. This result was supported by the study of Udomrati et al. (2020), who found that the water solubility index of RD41 (1.22%) was significantly lower than that of RD6 (1.72%), PT1 (1.68%), and KTH17 (1.42%). The lower leaching of starch molecules in RD41 might possibly be attributed to its high protein content (Table 1), which reduced water absorption by the starch granules due to competition in binding with water

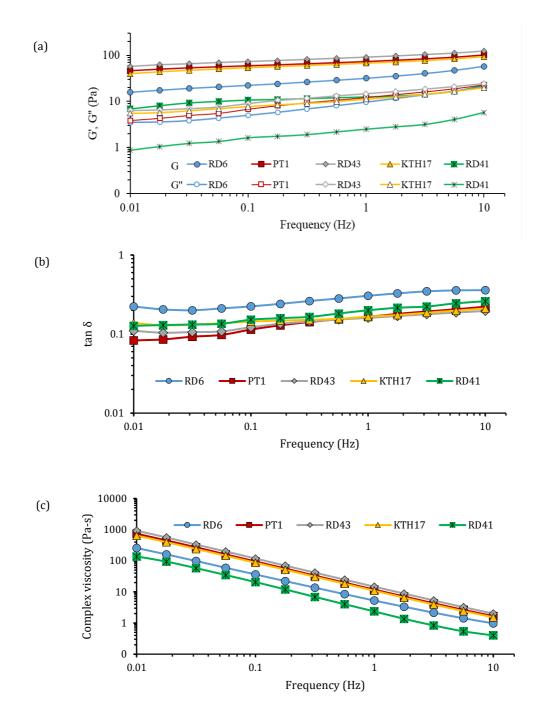
(Marco and Rosell, 2008). Proteins decrease the swelling and collapse of starch granules and the leaching of amylose molecules (Sun et al., 2008), resulting in both high *n* and low *k* values of RD41 (Table 2). Our result was in agreement with that of Lin et al. (2010), who reported that rice starch with higher amylose and protein contents exhibited good fluidity at a rice starch concentration of 8%. Moreover, lipid and amylose retard the water absorption by starch granules because their linear structure and their lipophilic part interrupt the binding of starch molecules with water (Chinma et al., 2015; Ali et al., 2016). Amylose and lipids could form an amylose-lipid complex, which inhibits the swelling of starch granules (Tester and Morrison, 1990) due to the reduction of the hydrophilic parts of starch molecules (BeMiller and Whisler, 2009). In addition, amylopectin reinforces the swelling power, water absorption index, and water solubility index of rice flour given that amylopectin enhances the water absorption ability of starch in crystalline areas and increases the amount of starch molecules that leach out (Ilowefah et al., 2015; Kraithong and Rawdkuen, 2019), leading to an increase in the viscosity of a system. The n values (0.12-0.15) of PT1, RD43, and KTH17 were quite similar, as shown in Table 2.

The LVR of all the rice pastes prepared from 5% w/w flour at 1 Hz frequency ranged from 0.01% to 10% strain, except for RD41 ( $\sim 0.01$ -1% strain; data not shown). In Figure 2a. G' was higher than G'' throughout the applied frequency range of 0.01-10 Hz. In all samples, G' and G'' were dependent on frequency because of the relaxation processes that occurred even within short time scales (Lopes da Silva and Rao, 1999), as seen in the less semi solid system. The less semi solid-like system allows for less



interconnection of starch molecules; hence, the junction zones in the system may have been easily destroyed, even if only a small frequency or shear rate is applied. Ahmed et al. (2008) reported that 4-8% glutinous rice flour was dependent on frequency, with a slope of 0.26-0.38, indicating less solid like characteristics. Less semi-solid-like behavior is indicated by tan  $\delta$  values higher than 0.1. In fact, the tan  $\delta$  values of RD6 and RD41 were the highest in this study and were higher than 0.1 throughout the experimental frequency range (Figure 2b); this is because the pastes of RD41 and RD6 had low and intermediate viscosity, respectively. The tan  $\delta$  values for PT1, RD43, and

KHT17 were approximately 0.1 at a low frequency because they all had a high viscosity paste, and their tan  $\delta$  values were dependent on frequency (Figure 2b). The tan  $\delta$  value (Figure 2b) seemed to be correlated to the n value (Table 2), with a decrease in the tan  $\delta$  value (increased solid-like behavior) as the n value decreased (increased shear-thinning behavior) as well, due to the increase in solid-like behavior formation with enhanced entanglement/interconnection and junction zones of starch molecules. The  $\eta^*$  of all samples decreased with increased frequency (Figure 2c), as is known based on static measurements (Brummer, 2006).



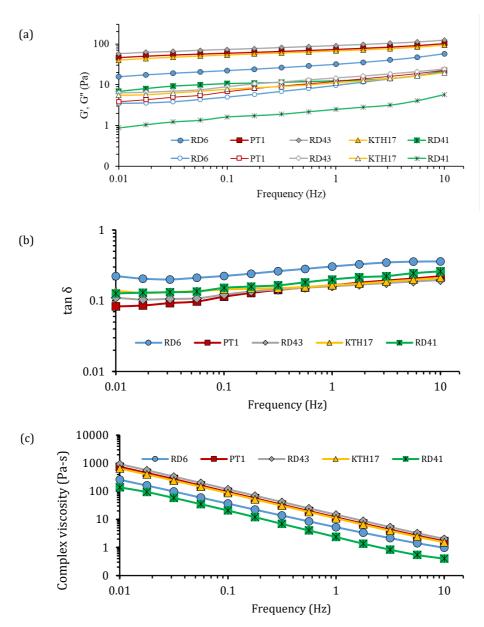
**Figure 2.** Frequency dependent oscillation profile of different varieties of 5% w/w gelatinized rice paste: (a) storage modulus (G') (solid symbol) and loss modulus (G') (clear symbol), (b) loss tangent (tan  $\delta$ ), and (c) complex viscosity ( $\eta$ ')



## 3.2.2 High rice flour concentration (gel system)

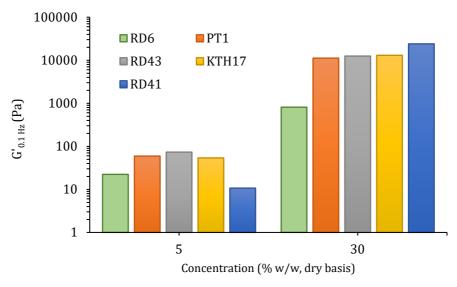
Rice flour concentration of 30% w/w was gelatinized (solgel transition) by heating the rheometer (25-95°C) and by cooling it to 25°C. When 0.01-10 Hz was applied to all the rice flour gels, G' was higher than G'' (Figure 3a); similar results were obtained for the system with 5% w/w flour (Figure 2a), indicating that both systems (5% and 30% w/w) were in the semi solid state. By contrast, G' is always less than G'' in a fluid system (Brummer, 2006). The G''value for the rice pastes prepared from high flour concentration (30% w/w)) was considerably higher (>4,000 times higher for RD41 and >200 times higher for PT1, RD43 and KTH17) than that for the rice pastes prepared from low flour concentration (5% w/w), as shown in Figure 4. The rigidity generated in the swollen granules at the flour concentration of 30% w/w was due to the limited water availability (Ahmed, 2010); consequently, an amylose network was formed, resulting in a firmer gel

and in a solid behavior. The difference between the G' and G'' values at a high flour concentration was greater ( $\sim 10$ times higher for RD6, ~200 times higher for PT1, RD43, and KTH17, and ~5,000 times higher for RD41) than that at the low concentration, as shown in Figure 2a and 3a, possibly because the increase in the flour concentration clearly involved a solid-like system (gel formation). The G' and *G*" values in the high flour concentration system were almost independent of frequency in all samples (except for RD6), as the structure of the strong gel was stable and was perhaps less disrupted during the application of low frequency or shear force. This result concurred with that of Ahmed et al. (2008), who found that a gel prepared from rice starch with a high concentration displayed solid-like characteristics, and the modulus of G' was independent of frequency (slope <0.07). The strong gel behavior of PT1, RD43, KTH17, and RD41 was confirmed by their tan  $\delta$ values, which were lower than 0.1 (Figure 3b).



**Figure 3.** Frequency dependent oscillation profiles of different varieties of gelatinized rice gel (30% w/w flour: (a) storage modulus (G') (solid symbol) and loss modulus (G'') (clear symbol), (b) loss tangent (tan  $\delta$ ), and (c) complex viscosity ( $\eta^*$ )

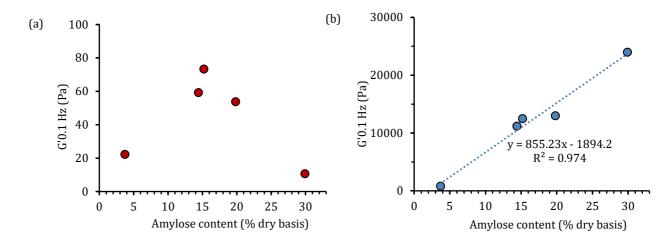




**Figure 4.**  $G'_{0.1\text{Hz}}$  of different varieties of gelatinized rice flour as a function of flour concentration of 5 and 30% w/w

G' indicates gel rigidity or gel strength. The G' value of RD41, which had the highest amylose and protein content (Table 1), was the greatest, as RD41 produced the strongest gel, possibly because amylose content was the main driving factor enhancing the cross-linking of network structure (Ye et al., 2016). Proteins may also enhance the hardness and intensity of a gel because the bonding of protein with amylose molecules forms a stiff network structure (Sun et al., 2008). Starch and protein macromolecules form a three-dimensional network that trap water molecules and swollen starch granules (Fradinho et al., 2018). Indeed, a protein network improves the stability of a rice flour gel (Lin et al., 2010). The tan  $\delta$  value of RD6 was higher than 0.1, indicating its being less solid-like and being a flowable paste, due to the considerably lower amount of amylose and the lack of retrogradation within a short-term period. The  $\eta^*$  of all samples decreased with increased frequency (Figure 3c), possibly because the structural deformation decayed with the increase in frequency (Ahmed, 2010).

There was no correlation between G' value and amylose content in the system prepared with 5% w/w rice flour (Figure 5a), as this concentration is possibly lower than the critical concentration ( $c^*$ ) for starch network formation; as a result, no gel was formed (flowable system). RD41 (30% amylose content) had the lowest G' because the water solubility index of RD41 was significantly lower than that of RD6, PT1, and KTH17 (Udomrati et al., 2020). Hence, RD41 was less viscous and exhibited a more liquid-like behavior in the diluted system. However, the relationship between amylose content and various rheological characteristics may be observed when the flour concentration is higher than the  $c^*$ value. Ahmed (2010) reported that the  $c^*$  of rice starch must be approximately 20% to provide a moderate gel network. When the rice flour concentration was 30% w/w, a positive linear correlation of G' with amylose content was observed, with an R<sup>2</sup> of 0.97 (Figure 5b). This relationship was supported by the findings of Biliaderis and Juliano (1993), who found in their study on rice starch (8-40%) that the influence of concentration on G' follows a power law correlation.



 $\textbf{Figure 5.} \textit{ G'}_{0.1\text{Hz}} \textit{ plot against amylose content of rice flour as a function of flour concentration: (a) 5\% w/w and (b) 30\% w/$ 



# 3.3 Rheological properties of high rice flour concentration (30% w/w) during gelatinization

# 3.3.1 Effect of temperature (25-95°C) on a system with high rice flour concentration

The oscillatory temperature sweep test was performed at 25-95°C. The G' and  $\eta^*$  values for RD6 and PT1 initially increased at 40°C because the starch granules began to swell slightly, as shown in Figure 6a and 6c, respectively. However, the G' and  $\eta^*$  values for KTH17 started to increase at the highest temperature ( $\sim$ 60°C), compared

with the other samples. This finding might have been caused by the denser and/or stronger crystallinity of the starch of granules in KTH17; such properties render the granules harder and exhibit less swelling at temperature belows the gelatinization temperature. The high density or strong crystalline structure or both of the KTH17 starch granules was confirmed by the highest  $\Delta H$  value and gelatinization temperature ( $T_0$ ,  $T_p$  and  $T_c$ ), as shown in Table 3.  $\Delta H$  and  $T_o$  indicate the quantity and strength of the crystalline structure of starch (Morrison and Azudin, 1987).

Table 3. Thermal properties for gelatinization of different varieties of rice flour

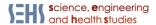
Rice variety	ΔH (J/g of flour)	T <sub>o</sub> (°C)	T <sub>p</sub> (°C)	T <sub>c</sub> (°C)
RD6	3.29a ± 0.04	$60.77^{d} \pm 0.14$	68.35° ± 0.35	79.04b ± 0.60
PT1	$3.34^a \pm 0.07$	63.33° ± 0.09	$68.74^{\circ} \pm 0.11$	$75.54^d \pm 0.11$
RD43	$3.42^a \pm 0.13$	$65.38^{b} \pm 0.07$	$71.07^{\rm b} \pm 0.11$	$77.69^{\circ} \pm 0.28$
KTH17	$3.56^a \pm 0.06$	$70.07^a \pm 0.37$	$75.32^a \pm 0.24$	$81.65^{a} \pm 0.20$
RD41	2.34b ± 0.17	56.27e ± 0.29	$66.34^{d} \pm 0.12$	72.15e ± 0.02

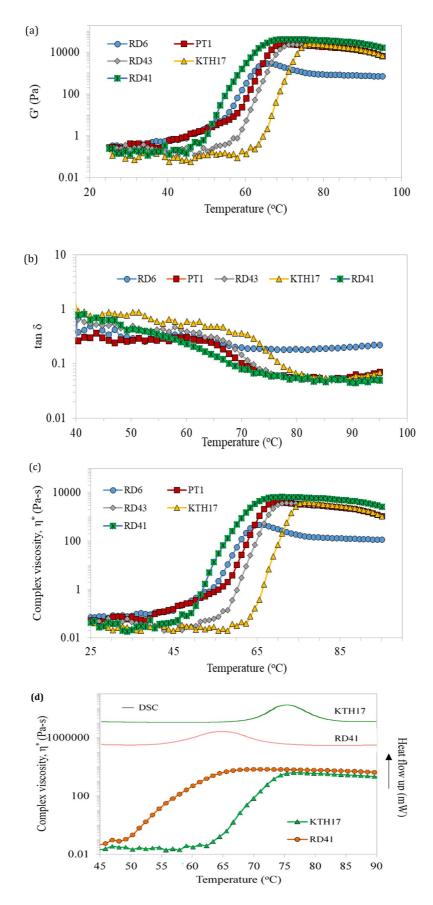
Note: \*Different letters (a,b,c,...) within a same column indicate significantly different at the 95% confidence level.

As the gelatinization temperature was reached, the G'and  $\eta^*$  values of all samples peaked (Figure 6a and 6c) because of the transition from sol (suspension) to solid (gel) due to the cross-linking of polymers (Isuka and Winter, 1994). During gelatinization, hydrogen bonding and some Van der Waals forces in the crystalline structure of starch were disrupted by heat. The water molecules moved into the starch granules and interacted to un bond the starch molecules with hydrogen bonding. The starch granules became swollen, growing much larger than their original size, resulting in an increase in volume fraction. The starch polymers (amylose and amylopectin) dissolved into the aqueous phase, leached out the interconnected amylose, and formed a threedimensional gel network in which the swollen starch particles interacted strongly, resulting in increased solidlike behavior (Eliasson, 1986; Hsu et al., 2000). As complete gelatinization occurred, the starch granules completely lost their crystalline structure, and swelling (weak swollen granules) was maximized. Figure 6d shows the DSC thermogram of gelatinization and  $\eta^*$ during heating (45-90°C) for KTH17 and RD41, showing the relationship between gelatinization temperature and the increase in  $\eta^*$  profile. Initially, the  $\eta^*$  value of RD41 rose steeply at low temperature (Figure 6d) because of its having the lowest gelatinization temperature (Table 3). This result might be attributed to the fact that starch granules of RD41 might exhibit a weaker crystalline structure and/or more damaged starch. Our previous research (Udomrati et al., 2020) showed that the starch damaged rate (%) was the highest for RD41 (5.78%) relative to the rates for RD6 (2.73%), PT1 (2.34%), RD43 (3.34%), and KTH17 (4.94%). The  $\eta^*$  value for KTH17 started to increase at a considerably high temperature, compared with that for RD41 (Figure 6d), probably because of the strong crystalline structure of the starch granules of KTH17. With a further increase in temperature, the G' and  $\eta^*$  values for all samples (excluding RD6) were quite constant (Figure 6a and 6c),

indicating irreversible swelling and solubilization of leached-out amylose (Biliaderis and Juliano, 1993). RD41 had a wider temperature range that corresponds to a constant *G'* value, compared with the rest of the samples, possibly because RD41 had the highest protein and lipid contents (Table 1), which could induce resistance to amylose leaching (Biliaderis et al., 1986). However, the  $\eta^*$  value for RD6 decreased immediately after complete gelatinization, possibly because its starchforming structure could be easily disintegrated and collapsed. The starch network might have contained softened, swollen granules, and there was less interaction between the granules and the network (Keetels and Van Vliet, 1994) due to the lack of amylose (almost none was present). At high temperatures (>90°C), the  $\eta^*$  values of all samples tended to decrease slightly (Figure 6c) because of the disintegration of the swollen granules (Rao, 1999). The G' value also tended to decrease at high temperatures (Figure 6a) because the starch granules were progressively weakened and softened due to the disentanglement of amylopectin molecules (Tsai et al., 1997). Also, the decrease in G' value might have been caused by the dynamic formation of an amylose gel matrix (Ahmed, 2010), and the gel network collapsed due to the loss of interaction between particles (Ahmed et al., 2008).

The tan  $\delta$  values of all samples were higher than 0.1 when the temperature was below the gelatinization temperature (Figure 6b) because the flour suspension (30% w/w) exhibited a liquid-like behavior. At temperature above the gelatinization temperature, the tan  $\delta$  values of all samples tended to continuously decrease because the gelatinized, swollen starch granules and the gel network formation of starch molecules improved the solid-like system. With a further increase in temperature, the tan  $\delta$  value remained quite constant because complete gelatinization had occurred. The tan  $\delta$  values for the completely gelatinized gel for all samples (except RD6) were lower than 0.1, indicating strong gel formation. RD6 formed a flowable paste (tan  $\delta$  >0.1).





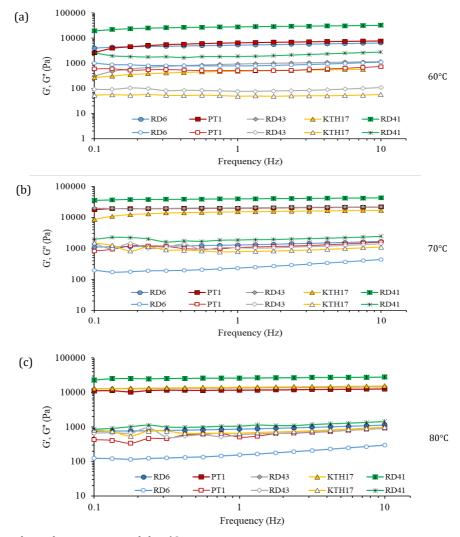
**Figure 6.** Temperature-dependent oscillation profiles of 30% w/w of different varieties of rice flour: (a) storage modulus (G'), (b) loss tangent (tan  $\delta$ ), (c) complex viscosity ( $\eta^*$ ), and (d) DSC thermogram compared with dynamic rheological data



# 3.3.2 Rice flour system with a high flour concentration at 60°C, 70°C and 80°C

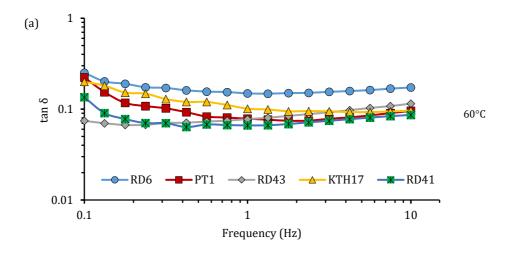
For all samples, the difference between the G' and G'' values at 60°C was narrower than that at 70°C and 80°C (Figure 7), and the tan  $\delta$  values for all samples were nearly 0.1 (Figure 8a). This finding might have been caused by the failure of the starch to completely gelatinize at 60°C; hence, the major system remained a suspension. The suspension system and the incomplete gelatinization at 60°C were confirmed by the opaqueness of the samples and the lack of gel formation after heating using a rheometer, as shown in Figure 10. At 70°C, the G' and G'' values for all samples (excluding RD6) tended to be higher than those at 60°C. This might have been caused by the gelatinized starch and by the strong gel formation, and this assumption was supported by the tan  $\delta$ values that were lower than 0.1 (Figure 8b). RD6 (glutinous rice) formed a less solid-like paste (tan  $\delta > 0.1$ ) because of the lack of an amylose-forming network. The difference between the tan  $\delta$  values at 70°C and 80°C for all samples was minimal (Figure 8b and 8C). At 80°C, the gel of all samples formed as a rice sheet (Figure 10) because complete gelatinization occurred, and there were many ordered amylose-forming gel structures; however, RD6 formed a flowable paste (Data not shown). The  $\eta^*$  values for all samples (excluding RD6) tended to increase as the heating temperature increased (Figure 9) because of the increased starch gelatinization. By contrast, the  $\eta^*$  value of RD6 decreased as temperature rose above 60°C to the higher temperatures (70-80°C), as shown in Figure 9, given that the swollen starch granules were weakened and disrupted, leading to the collapse of the gel network (Ahmed et al., 2008).

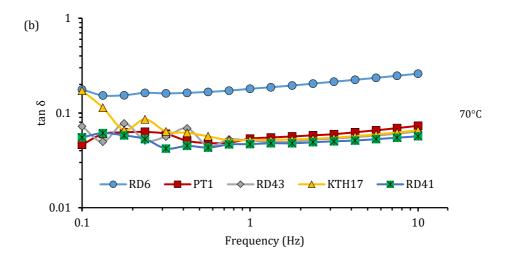
In terms of application, a hard gel could be formed using a high concentration of rice flour with a high amylose content or with a low to intermediate amylose content. A soft gel could be produced by using a low concentration of rice flour with a low to intermediate amylose content or by using mixed rice flours (such as glutinous rice flour mixed with rice flour with a high, intermediate, or low amylose content). Glutinous rice flour may be suitable for highly viscous and flowable food systems. The above rheological information about rice flour may be applied in selecting the appropriate material or ingredient, in designing processing equipment (e.g., mixer, piping equipment, and pumps), and in evaluating the stability of a product during processing, transportation, and storage.

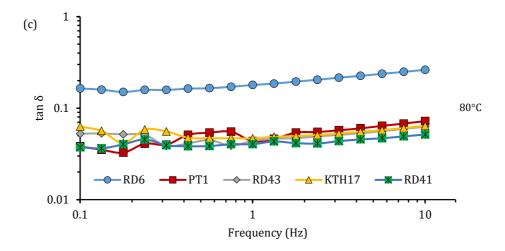


**Figure 7.** Frequency dependent storage modulus (G) (solid symbol) and loss modulus (G) (clear symbol) of different varieties of rice flour system (30% w/w flour) as a function of temperature: (a) 60°C, (b) 70°C, and (c) 80°C



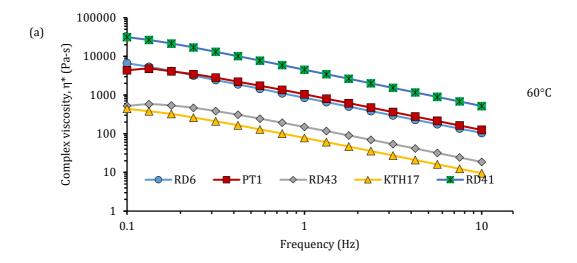


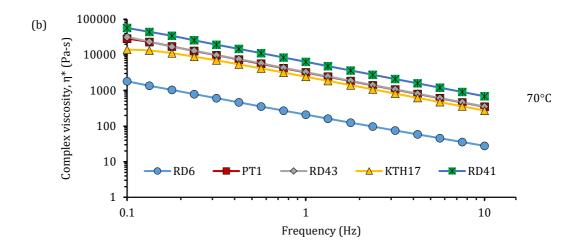


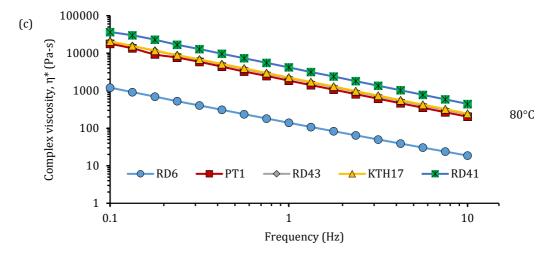


**Figure 8.** Frequency dependent loss tangent (tan  $\delta$ ) of different varieties of rice flour system (30% w/w flour) as a function of temperature: (a) 60°C, (b) 70°C, and (c) 80°C



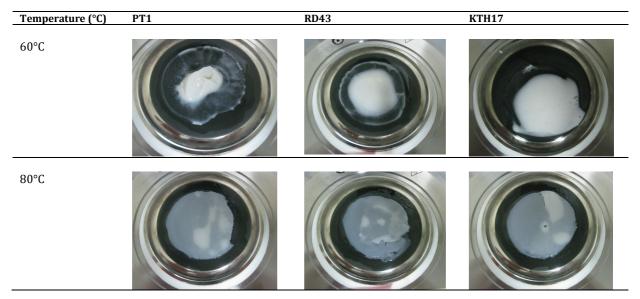






**Figure 9.** Frequency dependent complex viscosity ( $\eta^*$ ) of different varieties of rice flour system (30% w/w flour) as a function of temperature: (a) 60°C, (b) 70°C, and (c) 80°C





**Figure 10.** Appearance of different varieties of rice flour with concentration of 30% w/w after heating using a rheometer at different heating temperatures

## 5. CONCLUSION

Rheological information about rice flour is important in selecting the suitable flour to be used to achieve the desired characteristics of various food products and in estimating the stability of products. Rice variety, amylose content, and flour concentration affect the rheological behavior of rice flour. In this study, all gelatinized rice samples were flowable when a low concentration (5% w/w) of rice flour was used. The RD41 paste (with a high amylose content) had the lowest viscosity (~0.08 Pa.s). Viscous rice pastes were obtained using RD6 (glutinous), PT1 and RD43 (with a low amylose content), and KTH17 (with an intermediate amylose content), and their viscosity ranged from 0.2 to 0.8 Pa.s. All the rice flours formed a gel network at a high concentration (30% w/w), except RD6 (glutinous), which formed into a highly viscous flowable paste. The hardness of the rice gels was strongly correlated with amylose content. The structure of all rice gel samples (except RD6) was less disrupted when a low frequency was applied, and this characteristic is related to stability during storage.

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