

# Effects of Product Temperature and Moisture Content on Viscoelastic Properties of Glutinous Rice Extrudates

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## ABSTRACT

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The viscoelastic properties of glutinous rice flour extruded at moisture contents of 45–55% and barrel temperatures of 75–95°C have been investigated using a small amplitude oscillatory rheometer. High moisture contents (50 and 55%) resulted in product temperatures 3–5°C lower than the barrel temperatures. It appeared that the moisture content was a key element in influencing the value of  $G'$  and  $\tan \delta$ . Raising product tem-

perature reduced the difference in  $G'$  caused by the moisture content. When the product temperature was >85°C, the extrudates yielded a similar degree of gelatinization despite the difference in moisture content. Meanwhile, both  $G'$  and  $G''$  decreased due to the disintegration of starch granules. The relationship between the energy input, measured as specific mechanical energy, and the viscoelastic properties was also assessed.

*Mochi*, made from glutinous rice, is a popular product in Oriental areas, including Taiwan, China, Japan, and others. The product exhibits unique viscoelastic properties that attract consumer attention. Conventionally, polished glutinous rice (*Oryza sativa*) (also known as waxy or sweet rice) is washed, cooked with water, then pounded to lose the integrity of the rice kernel to become *mochi*. Sugars or other ingredients, such as peanut powder, can be used to adjust the taste and flavor. The traditional method is a batch process, which is labor-intensive and time-consuming due to cooking and pounding. The stickiness of cooked glutinous rice creates handling difficulties. Extrusion cooking integrates mixing, cooking, kneading, and forming into one piece of equipment and appears to be an attractive method for manufacturing *mochi*. In addition, the extruded product can be further dried and ground to prepare precooked rice flour. Before applying extrusion technology to these products, the properties of extruded products need to be understood.

Extrusion cooking of rice flour appears to be a simple process, but the control of finished product characteristics is rather complicated. Both temperature and concentration of water are key elements affecting the internal structure as well as the viscoelastic properties of extruded cereals (Brent et al 1997). Increasing barrel temperature and screw speed generally decreased peak viscosity, hot-paste viscosity, and cold-paste viscosity of rice extrudates as measured by Rapid Visco Analyser (RVA) (Guha et al 1998). Die temperature was another important parameter influencing the viscosity of rice starch during extrusion (Akdogan et al 1997). Horiuchi (1980) pointed out that the optimum concentration of solid matter was  $\approx 45\%$  for making *mochi*. The literature related to extrusion cooking using a moisture content that high is limited. Specific mechanical energy (SME) has been frequently used to quantify the energy input from the conversion of mechanical to thermal energy. SME decreases with the ratio of feed rate to screw speed and also is a function of die temperature (Yeh and Jaw 1999). SME, product temperature, and residence time have been used to predict the properties of puffed products such as expansion index and sensory characteristics (Meuser et al 1986) and solubility (van Lengerich 1984, Pfaller and Meuser 1988). The relationship between SME and viscoelastic properties of extrudates with high moisture content would be helpful for designing operating conditions to prepare various products such as precooked pasta, third-generation snacks, and *mochi*.

Small amplitude oscillatory shear (SAOS) is an appropriate tool to illustrate the viscoelastic behavior of a material. The response of

a system can be resolved into elastic ( $G'$ ) and viscous ( $G''$ ) components, which provide a better understanding of the behavior of starch gels (Eliasson 1986) and extruded cereal melts (Brent et al 1997). Moisture content is an important element in determining the viscoelastic properties of cereals. Water can act as inert filler, causing the dynamic properties to reduce proportionally to moisture content or behave as a lubricant, enhancing the relaxation phenomena (Masi et al 1998). Cooking a starch dispersion at high temperature (95°C) resulted in a paste with an extensive network structure and low  $G'$ . When the cooking temperature was lowered to 70°C, the rigidity ( $G'$ ) was increased due to the presence of swollen starch granules but the paste lacked the network structure. The SAOS measurements can be used to differentiate the properties of cooked corn starch and oat flour gels (Shim and Mulvaney 1999). Amylose content also affects the dynamic response of nonwaxy rice flour paste. Rice flour with high amylose content gave a paste with high elasticity (Reddy et al 1994). The application of SAOS to characterize the viscoelastic properties of *mochi* may be helpful for optimizing the manufacturing process.

Generally, the operation of a single-screw extruder is limited to low moisture content due to wall slip problem. However, it is an attractive tool in terms of cost of production. This work was intended to evaluate the feasibility of processing glutinous rice flour at high moisture content using a single-screw extruder. We also attempted to investigate the effects of product temperature and moisture content on the viscoelastic properties of extrudates. The result should be helpful for understanding the material properties in the rubbery region.

## MATERIALS AND METHODS

### Material

Polished glutinous rice (first crop of Taichung Waxy 70 in 1998) was purchased from Fen-Yuan Agriculture Association in Central Taiwan. The rice was ground using a stamp mill to pass a 100-mesh (ASTM) screen. The proximate composition was analyzed by AOAC methods (1984). The content of total starch was analyzed according to the method of Chiang and Johnson (1977). An enzymatic method (Yun and Matheson 1990) was employed to determine the amylose content. Rice flour was put in a silent cutter and distilled water was sprayed on the rice flour to yield a moisture content of 40% (wb). The silent cutter not only provided the mixing, but also prevented the rice from caking. Therefore, the moisture distribution in rice flour was uniform. The preconditioned rice flour was stored at 4°C until used.

### Extrusion

The extrusion cooking was conducted in triplicate by using a split-barrel single-screw extruder (Tai-Yu Co., Taipei, Taiwan) as described by Yeh and Jaw (1998). The bore of the profiled barrel

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had a diameter of 8.5 cm. The L/D ratio was 5.1:1. A torque transducer (TP-10KMCB, Kyowa Co., Ltd., Tokyo) was fitted between the motor and gear box to measure the torque. A dynamic strain amplifier (DPM-700B, Kyowa) was used to amplify the signal, which was recorded by a personal computer. The energy losses in the bearing and the drive train were assumed to be negligible because they did not affect the reproducibility of the control system. The feature of the screw profile was to use mixing disks. Ten one-tipped paddle type mixing discs (Yeh and Jaw 1999) with a unit length of 1 cm were staggered at 60° in the reverse direction. There were two barrel sections, jacketed and nonjacketed. The jacketed barrel was 29.5 cm long, measured from the feeding port. Water flowed inside the jacket for cooling. A heating tape (8 cm wide) around the nonjacketed barrel (2 cm away from the jacketed barrel) was used for heating. There was a thermocouple attached on the surface of the nonjacketed barrel to measure the barrel temperature. The heating tape and water flow were on-off controlled to keep the nonjacketed barrel at a set point of 75, 80, 85, 90, and 95 ± 2°C. The circular die opening had a diameter of 10 mm. A thermocouple was inserted into the die to measure the product temperature.

From preliminary tests, a screw speed of 80 rpm and feed rate of 8 ± 0.3 kg/hr were selected. The extruder was run under starved conditions in this study. Preconditioned rice flour was fed into the extruder through a twin-screw feeder that was calibrated before each experiment. During the experiments, the rice flour in the hopper of the feeder was controlled manually at a given level (≈5 cm above the screw). Thus, feeding was kept smooth and consistent with a deviation of ±0.3 kg/hr. When the preconditioned rice flour was fed into the extruder, water was also pumped into the extruder to produce an in-barrel moisture content of 45, 50, and 55 ± 1%. After stable operations were established, judged by constant torque readings and product output, extrudates were collected for further analysis. The extrudate was dough-like and very sticky. There was no internal bubbling or puffing under the operating conditions selected.

### SME

No-load torque was measured while operating with flood feeding of water. The no-load torque was subtracted from the operational torque to obtain the corrected torque. The SME was calculated using the equation:

$$\text{SME} = [(2\pi \times \tau \times S_s/60)/F_T] \times 3.6 \text{ [kJ/kg]} \quad (1)$$

where  $\tau$  is corrected torque (N·m),  $S_s$  is screw speed (rpm), and  $F_T$  is feed rate (kg/hr) and equivalent to the product output at stable conditions.

### Thermal Analysis

The gelatinization behavior of glutinous rice flour with different moisture contents (40–90%) was analyzed using differential scanning calorimetry (Setaram DSC 121; Setaram Co., France) equipped with a liquid-nitrogen intercooler. Water was added to the rice flour to the desired moisture content. The mixture was hermetically sealed in a stainless steel crucible and stored in a refrigerator (4°C) overnight for uniform moisture distribution. An empty crucible was used as the reference. The scanning temperature was increased from 30 to 145°C at 5°C/min. The onset ( $T_o$ ), peak ( $T_p$ ), and conclusion temperature ( $T_c$ ) and heat of gelatinization were determined using the analysis software provided by Setaram Co.

DSC was also used to determine the degree of gelatinization of the extrudate in triplicate according to the method of Wang et al (1989). The extrudate was vacuum-dried at 40°C for 48 hr, then ground to pass a 100-mesh screen. The powder was adjusted to the corresponding in-barrel moisture content. The preparation of samples and DSC operation were the same. The heat of gelatinization ( $\Delta H_T$ ) was determined by using a DSC data-analysis system provided by Setaram. The heat of gelatinization of raw material

with the same moisture content was designated as  $\Delta H_o$ . The degree of gelatinization (DG) was calculated as:

$$\text{DG} = (\Delta H_o - \Delta H_T) / \Delta H_o \times 100\% \quad (2)$$

### SAOS of Rice Flour-Water Mixtures

The viscoelastic properties of samples were measured using a controlled stress rheometer (Carri-Med CSL 500, TA Instruments Ltd., England) with parallel-plate geometry of 40-mm diameter and a gap of 3 mm. Linear range at a strain of 0.2% and a frequency of 1 Hz, as determined from the preliminary tests, was used in this study. Silicone oil coating the outer edge of the sample and a solvent trap were employed to minimize the water loss during measurements. A temperature sweep was conducted in triplicate for the rice flour with moisture content of 45, 50, and 55%. The temperature was 25–95°C at a heating rate of 2.5°C/min. Three parameters, storage modulus ( $G'$ ), loss modulus ( $G''$ ), and loss tangent ( $\tan \delta$ ), were obtained using the software of the analysis program and the averaged values of the parameters were reported.

### SAOS of Extruded Samples

When the extruder was at a stable condition, the extrudate was collected immediately at the exit of die and sealed in a double-layered PVC bag to reduce the moisture loss. The sample was cooled to room temperature (25°C) in 12 hr. The moisture content of cooled extrudate was double-checked using AOAC methods (1984). The deviation of moisture content was ±0.8%. It was reasonable to neglect the water loss after extrusion. A rheometer was again used to analyze the viscoelastic property of extrudate at 25 ± 0.1°C in triplicate at a strain of 0.2% and frequency of 1 Hz. Silicone oil was also applied to avoid the moisture loss. As for the rice flour, the average values of  $G'$ ,  $G''$ , and  $\tan \delta$  were obtained. In addition, moisture contents and dynamic rheological properties of five commercial products of *mochi* prepared by traditional methods were also analyzed by these procedures.

### Water Soluble Index

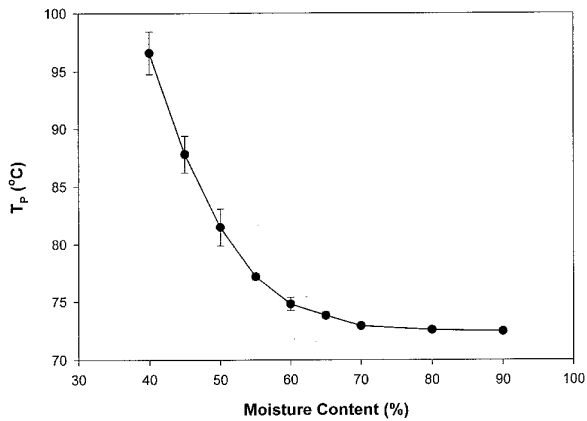
Water soluble index (WSI) was determined with 0.1 g of dry extrudate powder using a modified method of Tsai et al (1997). Extrudate powder was weighed into a centrifuge tube with a coated screw cap, to which 10 mL of distilled water was added. The tube was put in a shaking water bath at 25°C for 1 hr. Then, the tube was centrifuged (8,000 × g) for 20 min. The supernatant (5 mL) was obtained by using a pipette (Biohit, Proline Co., Finland) and dried to constant weight ( $W_1$ ) in an air oven at 105°C. The determination of WSI was conducted in triplicate and the average was recorded.

$$\text{WSI} = [W_1 \times 2/0.1] \times 100\% \quad (3)$$

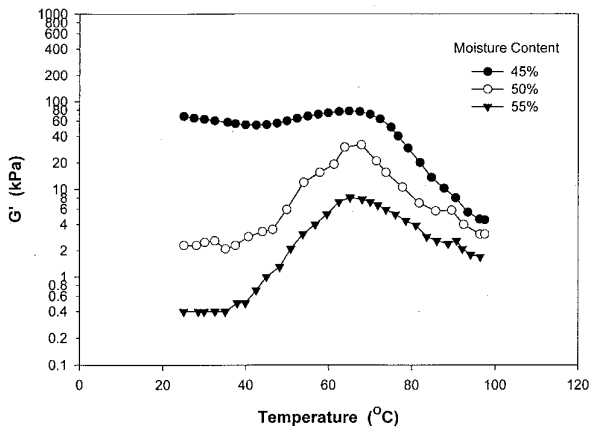
## RESULTS AND DISCUSSION

The rice flour was composed of water (10.94 ± 0.09%); crude protein (7.41 ± 0.07%); crude lipid (0.52 ± 0.02%); ash (0.35 ± 0.01%); and total carbohydrate (80.78 ± 0.58%). The total starch content was 88.97 ± 3.61% (db), with an amylose content of 2.51 ± 0.54% (based on the dry weight of starch), which was lower than the data (5.93%) reported by Noomhorm et al (1997). Product temperatures of extrudates with different in-barrel moisture contents at various barrel temperatures are shown in Table I. The product temperatures were about the same as the barrel temperatures when the in-barrel moisture was 45%. As the in-barrel moisture increased to 50 and 55%, the product temperatures were 3–5°C lower than the barrel temperature. This might result from the cooling effect of moisture.

The gelatinization behavior of rice flour was observed by using DSC and SAOS. From the DSC thermogram,  $T_o$  was ≈64.5 ± 1.5°C, which was lower than the 70°C mentioned by Noomhorm



**Fig. 1.** Differential scanning calorimetry peak temperature ( $T_p$ ) of rice flour as a function of moisture content.



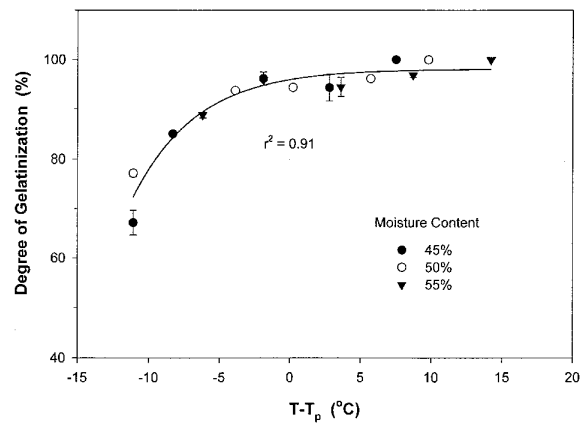
**Fig. 2.** Temperature sweep of rice flour with different moisture contents at 1 Hz.

**TABLE I**  
Product Temperatures Measured at Various Barrel Temperatures and Moisture Contents

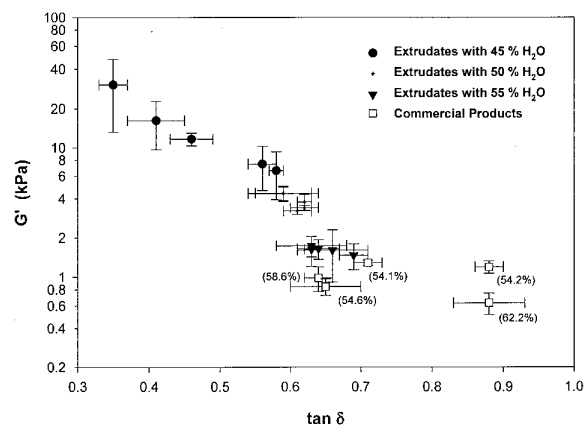
Barrel (°C)	Product (°C) at In-Barrel Moisture Content		
	45%	50%	55%
75	76.7 ± 0.8	70.4 ± 0.8	71.0 ± 2.8
80	79.5 ± 1.0	77.6 ± 2.6	75.3 ± 3.1
85	85.9 ± 1.1	81.7 ± 2.1	80.8 ± 3.0
90	90.6 ± 1.7	87.2 ± 2.5	85.9 ± 3.7
95	95.3 ± 0.7	91.3 ± 2.9	91.4 ± 2.6

et al (1997). It appeared that  $T_0$  was not a function of moisture content variation at 40–90%. The  $T_p$  was dependent on moisture content, as illustrated in Fig. 1. The  $T_p$  decreased linearly as the moisture content increased from 40 to 60%. When the moisture content was >60%,  $T_p$  reached a plateau value of  $72.9 \pm 0.9^\circ\text{C}$ . The observation was similar to the report by Wang et al (1989) for corn starch. The second endothermic peak observed for low moisture content (Biliaderis et al 1986) was not detected here due to the fact that the moisture content was >40%. DSC thermograms showed a peak corresponding to the amylose-lipid complex between 110 and 120°C.

The moisture content significantly affected  $G'$  of glutinous rice flour (Fig. 2). Due to the compactness of rice flour, the sample with 45% moisture yielded the highest initial  $G'$  (65.7 kPa), which was 322× that of the sample with 55% moisture. This is in agreement with the concept that the concentration of solid matter is a key element in determining viscoelastic properties of rice pastes (Reddy et al 1994). Heating from 25 to 65°C resulted in the swelling of



**Fig. 3.** Relationships between degree of gelatinization and  $T - T_p$ ;  $T$  was the product temperature of the extrudate and  $T_p$  was the differential scanning calorimetry peak temperature at corresponding in-barrel moisture.

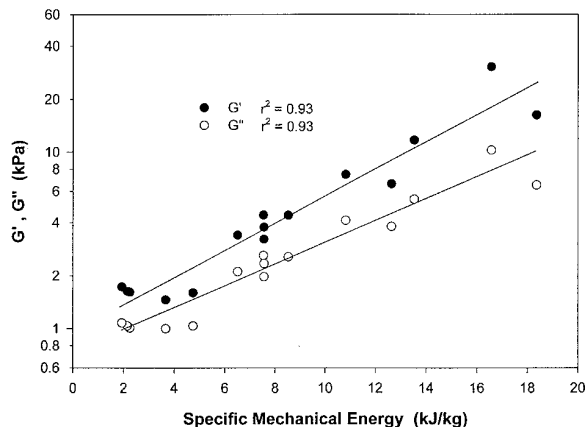


**Fig. 4.** Viscoelastic properties ( $G'$ ,  $\tan \delta$ ) of extrudates and commercial products. Values in parentheses are moisture content of commercial product. Both  $G'$  and  $\tan \delta$  are measured values; error bars are shown for both parameters.

starch granules and the increase in  $G'$  of samples with 50 and 55% moisture. However, heating did not cause significant increase in  $G'$  of the sample with 45% moisture. This might have resulted from the restricted swelling of rice starch granules at this high concentration of solid matter. Continuous heating resulted in the disintegration of starch granules and a decrease in  $G'$ . The final  $G'$  at 95°C varied from 4.9 to 2.0 kPa as the moisture content was increased from 45 to 55%. This implied that the disintegration of starch granules reduced the effect of moisture content on  $G'$ . Thus, the double reduction procedure that accounted for the moisture effect on viscoelastic properties of wheat flour at different frequencies (Masi et al 1998) is not applicable in this study. There was a maximum  $G'$  ( $G'_{\max}$ ) during the temperature sweep. The sample with 45 and 55% moisture exhibited  $G'_{\max}$  of 83.7 and 8.7 kPa, respectively. The temperature corresponding to  $G'_{\max}$  temperature was designated as  $T_{G'_{\max}}$ . It is worth noting that the variation of  $T_{G'_{\max}}$  was in a narrow range (65.0–66.3°C). Apparently, the moisture content significantly affected neither  $T_{G'_{\max}}$  nor  $T_0$  of the DSC thermogram.

#### Extruded Samples

Both product temperature and in-barrel moisture content affected the degree of gelatinization of extrudate. In general, both high in-barrel moisture and high product temperature resulted in extrudates with a high degree of gelatinization. The degree of gelatinization appears to be influenced ( $r^2 = 0.91$ ) by the difference between

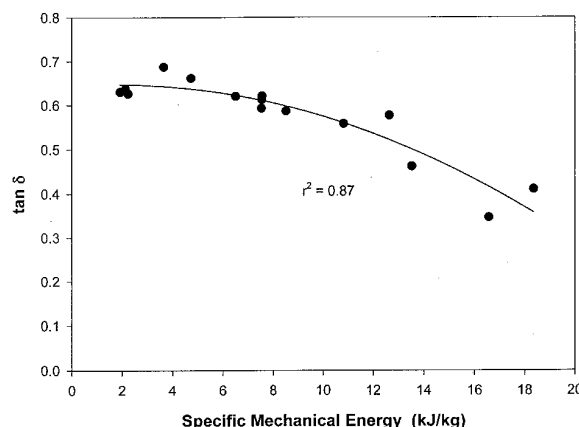


**Fig. 5.** Viscoelastic properties ( $G'$  and  $G''$ ) of extrudates as functions of specific mechanical energy.

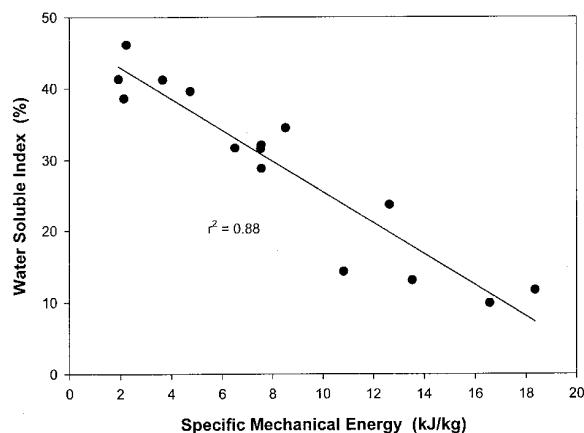
product temperature ( $T$ ) and DSC peak temperature ( $T_p$ ) at the corresponding moisture content (Fig. 3). When  $T - T_p$  was  $< -5^\circ\text{C}$ , the degree of gelatinization was lower than 90%. The degree of gelatinization reached 100% when  $T - T_p$  was  $> 7.5^\circ\text{C}$ , except for the extrudate with product temperature of  $85.9^\circ\text{C}$  and in-barrel moisture of 55%.  $T_p$  accounted for the effect of moisture and appeared to be a good reference temperature for discussing the effect of processing on the degree of cooking. DSC analysis showed starch completed gelatinization at temperatures  $> 100^\circ\text{C}$  when the in-barrel moisture content was  $< 55\%$ . As reported by Shim and Mulvaney (1999), higher stirring speed from RVA shifted the viscosity of corn starch dispersions to lower values due to the disintegration of starch granules. The rotation of the screw element would exert more disintegration effect on starch granules. Thus, extrudates exhibited 100% degree of gelatinization at  $T - T_p < 15^\circ\text{C}$ .

The absolute value of  $G'$  for extruded samples (Fig. 4) was smaller than that for the corresponding rice paste of the same moisture content and temperature in Fig. 2. This might be due to additional disintegration of starch granules after extrusion cooking. The balance between the fraction of starch granules and formation of the paste contributes the value of  $G'$  (Shim and Mulvaney 1999). For the extrudate with 45% moisture, heating at  $> 80^\circ\text{C}$  resulted in the disintegration of starch granules, a decrease in  $G'$ , and an increase in  $\tan \delta$ . In other words, the disintegration of starch granules resulted in materials with more liquid-like behavior. The extrudate at  $79.5^\circ\text{C}$  exhibited the most solid-like behavior as indicated by the lowest value of  $\tan \delta$ . The increase in moisture content caused a reduction in  $G'$  and an increase in  $\tan \delta$ . Nevertheless, the change in product temperature caused a slight variation in  $G'$  of extrudates with 50 and 55% moisture. All of the commercial products tested yielded  $G'$  of 0.6–1.3 kPa and  $\tan \delta$  of 0.6–0.9. Compared with the commercial products with moisture of 54–55%, the extrudates with 55% moisture yielded slightly higher  $G'$  and lower  $\tan \delta$ . It is not surprising that the commercial products with moisture content of 62.2% yielded low  $G'$  (0.63 kPa) and high  $\tan \delta$  (0.88). Single-screw extrusion cooking with high moisture content is a feasible process for preparing *mochi* (Fig. 4). However, the extrudates tended to exhibit more solid-like behavior than the commercial products, indicated by higher  $G'$  and lower  $\tan \delta$ , probably due to the kneading exerted by the rotation of screw.

The extrusion cooking was operated with high moisture and large die opening; therefore, the SME was  $< 20$  kJ/kg, which was lower than SME for extrusion of pasta (27–122 kJ/kg) (Abecassis et al 1994). The SME also was lower than that for twin-screw extrusion of rice starch at moisture contents of 57–65% (Akdogan et al 1997). The difference resulted from the die opening area ( $78.54$  mm<sup>2</sup>) used in this study, which was much larger than that ( $29.4$  mm<sup>2</sup>) used by Akdogan et al (1997). According to the classification by Harper (1989), the extruder used in this study can be considered a



**Fig. 6.** Effect of specific mechanical energy on viscoelastic property ( $\tan \delta$ ) of extrudates.



**Fig. 7.** Water soluble index of extrudates as a function of specific mechanical energy.

low-shear cooking extruder. High moisture and high temperature resulted in low viscosity, which required less energy input during extrusion cooking. Both  $G'$  and  $G''$  of extrudates are power law functions of SME (Fig. 5). High  $G'$  and  $G''$  were associated with high SME. This is similar to the finding that high apparent viscosity is associated with high SME during extrusion cooking of rice starch (Akdogan et al 1997). That  $G'$  was always higher than  $G''$  implied the formation of a weak gel. Higher energy input resulted in extrudates with more solid-like behavior, as indicated by the decrease in  $\tan \delta$  (Fig. 6). Although increasing SME causes degradation of starch molecules, indicated by a decrease in intrinsic viscosity (Yeh and Jaw 1999), the degradation of starch molecules in our experiments could be assumed to be negligible due to high moisture and large die openings. Thus, proper input of mechanical energy enhanced the strength of structure of extrudate indicated by the increase in  $G'$ . This might be the reason why pounding is necessary in the traditional method. In other words, the solid was held tight, and less solid dissolved in water. Therefore, WSI decreased linearly ( $r^2 = 0.88$ ) as the SME increased (Fig. 7). The results demonstrate that the rotation of screw elements enhanced the structure strength of glutinous rice extrudate, which yielded viscoelastic properties similar to *mochi* prepared by the traditional method.

## CONCLUSIONS

Glutinous rice extrudates with 45–55% moisture formed weak gels and exhibited viscoelastic properties. Moisture content and product temperature were two key elements which influenced the extrudate properties as determined by  $G'$  and  $G''$ . Increase in SME resulted in an increase in both  $G'$  and  $G''$  and a decrease in  $\tan \delta$ ,

which indicated an increase in solid-like behavior. The WSI was reduced by increasing SME. It appeared that an increase in SME enhanced the strength of extrudate structure. The results demonstrate that single-screw extrusion cooking at high moisture content is feasible to prepare *mochi*.

#### ACKNOWLEDGMENTS

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