Flow Properties of Low-Moisturized Starch Melts at an Elevated Temperature

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The flow properties of starch melts (potato and corn starches) at the moisture content of 16, 20, 25, 30 and 35% (dry basis) were characterized using a capillary tube viscometer. By use of measured flow rates (Q, m³ s⁻¹), and pressure drops of capillary tube (ΔP_c, MPa) and orifice (ΔP_o, MPa), the relationships between the flow rate and the pressure drop in the capillary tube (ΔP_d, MPa) were obtained. The flow curves of both starch melts showed good linearly on logarithmic scales. These starch melts, however, gave a yield stress, especially at low moisture content. Then both flow curves satisfied an equation by Herschel-Bulkley power-law model after exchanging the flow rate and the pressure drop to apparent shear rate ($\dot{\gamma}_a$, s⁻¹) and shear stress (τ_w, MPa), respectively. The flow properties were good representative by Herschel-Bulkley power-law model and these curves varied with the moisture content. The flow properties between potato and corn starch melts were different even though they were same moisture content, especially that was reflected in the consistency index.

INTRODUCTION

Extrusion cooking is widely used to manufacture foods and feeds from cereal, tuber or other protein/carbohydrate/water mixtures, which are known generally as food polymers. The extrusion cooking is usually carried out under the conditions of high temperature and high pressure. Under these conditions low moisture food polymers can be fused. The flow properties of food polymer melts have been studied using an extruder to help for engineering design and scale up (Cervone & Harper, 1978; Chen *et al.*, 1978; Jao *et al.*, 1978; Bhattacharya & Hanna, 1986). Although these investigations provided important information about extrusion cooking, further fundamental studies on the properties of food polymer melts were considered necessary in order to obtain basic information about thermo-mechanical properties. Fujio *et al.* (1991) and Hayashi *et al.* (1991, 1993) studied the flow properties of soy protein isolate melt at a high temperature (140 °C) using a capillary tube viscometer. It is necessary for analyzing the further flow properties, however, that the end effects occurred through a capillary tube are corrected. Bagley's end correction method (Bagley, 1957) is usually used for the correction. However the operation of Bagley's end correction method was complicated. Therefore Hayashi *et al.* (1991) developed an other correction method using orifice and confirmed the availability of this method.

In this paper we try to elucidate the flow properties of low moisturized potato and corn starch melts by use of orifice correction method and to fit the flow properties to a power-law model by regression analysis.

MATERIALS AND METHODS

Starches

Potato and corn starches were purchased from Nacalai Tesque Co. (Tokyo, Japan; potato, Lot No. M1E-5704; corn, Lot No. M1H-9367). After these starches were dried at 70°C for 48 hour, the moisture content of the dried starches was adjusted to 16%, 20%, 25%, 30% and 35% (dry basis) by blending with desired amount of fine ice powder (ca. 20 mesh pass; at −20°C) in a cold room at −20°C. After drying at 105°C for 10 hour, the moisture content was defined as zero. The moisture-adjusted starches were stored in a cold room at 4°C until use.

Flow properties

A cross-sectional view of a capillary tube viscometer (Shinmeiwa Co. Ltd. Tokyo, Japan) using for the measurement of the flow properties of starch melts is shown in Fig. 1. The detail of the viscometer has been described in Hayashi *et al.* (1990) and Fujio *et al.* (1991). A 4.0 g of the moisture-adjusted starch was moulded into a cylindrical shape (1.08 cm in diameter and about 3.0 cm in length) using a hand press (Shimadzu Co. Ltd., Kyoto, Japan) and placed into the sample reservoir. The reservoir was heated to 150°C in advance and the measurements of flow properties were performed at 150°C, at which the starches melt sufficiently (Igura *et al.*, 1996). Under the condition of vertical load at 15 MPa, the starch was kept for 15 min in the reservoir for melt them. Two types of the die were used for the measurements of flow properties in the capillary tube. The one was a
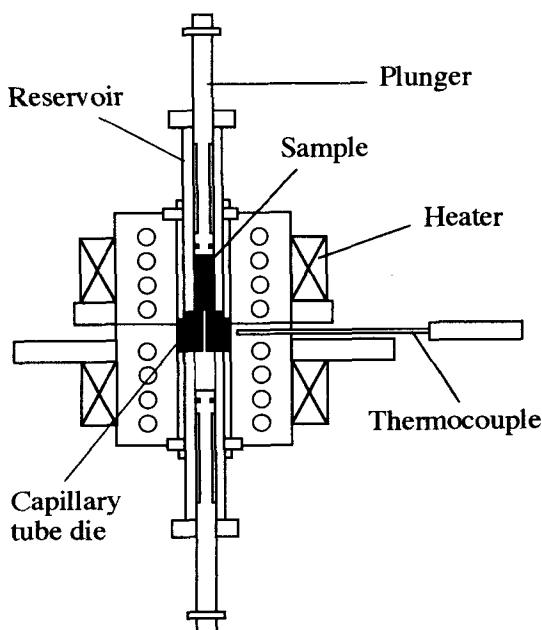

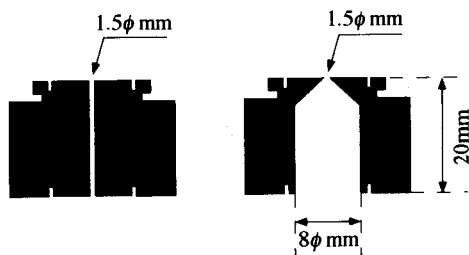**Fig. 1.** Cross-sectional view of capillary tube viscometer.

Fig. 2. Designs of capillary tube (left) and orifice (right).

capillary tube which has 0.75 mm in radius (R) and 20 mm in length (L), and the other was an orifice which has same radius as the capillary tube (Fig. 2). The resultant pressure drops were measured for a capillary tube (ΔP_c, MPa) or an orifice (ΔP_o, MPa) at various plunger speeds, i.e. various volumetric flow rate, Q (m³ s⁻¹). End effect correction was done by subtracting ΔP_o from ΔP_c at the same value of Q. This gives ΔP_d ($= \Delta P_c - \Delta P_o$) which indicates the pressure drop in the capillary tube. The volumetric flow rate, Q, was converted into apparent shear rate ($\dot{\gamma}_a$, s⁻¹) by using the equation: $\dot{\gamma}_a = 4Q/\pi R^3$. The pressure drop in the capillary tube, ΔP_d, was converted into shear stress by using the equation: $\tau_w = \Delta P_d \times R/2L$.

Regression analysis

The Herschel-Bulkley power-law model (Skelland, 1967) was applied to measured data in order to characterize the flow properties of starch melts:

$$\tau_w = \tau_0 + (\eta' \times \dot{\gamma})^n$$

where τ_0 (MPa) is the yield stress, i.e. the minimum shear stress required for flow; n (dimensionless) is the flow behaviour index; η' (MPa$^{1/n}$ s) is the consistency index.

Since the model equation is intrinsically non-linear, the ordinary regression method for linear equations is not applicable. Therefore, the successive approximative calculation was performed iteratively at each moisture content using a computer (PC-9801DS, Nippon Electric Co. Ltd., Tokyo, Japan) to find the regression coefficients that minimize the residual sum of squares (Snedecor & Cochran, 1972).

RESULTS AND DISCUSSION

Flow curves of starch melts

Figure 3 and 4 show the observed volumetric flow rates, Q (m³ s⁻¹), and resultant ΔP_c or ΔP_o (MPa) on logarithmic scales of potato and corn starch melts, respectively. The pressure drop of the starch melts increased linearly as increasing the flow rate. These relationships between flow rates and pressure drops changed as the moisture content was changed. To further analyze the flow properties of these starches, the pressure drops in the capillary tube, $\Delta P_d = \Delta P_c - \Delta P_o$, were calculated at same flow rates applying the correction method using orifice (Hayashi *et al.* 1991). The relationships between
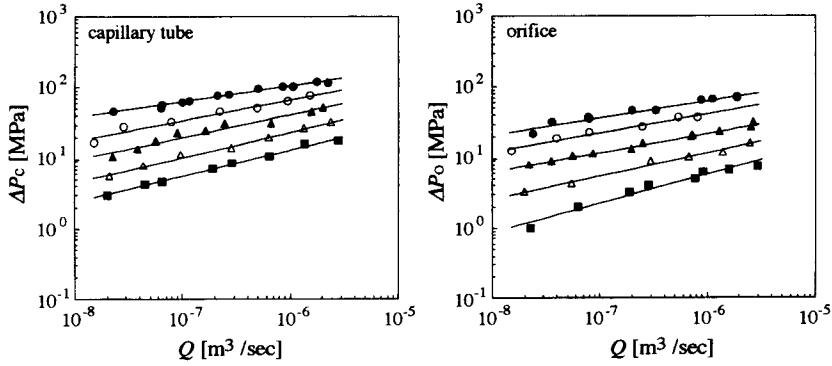
Fig. 3. Relationships between flow rate and pressure drop for potato starch melts with various moisture content at 150 °C.
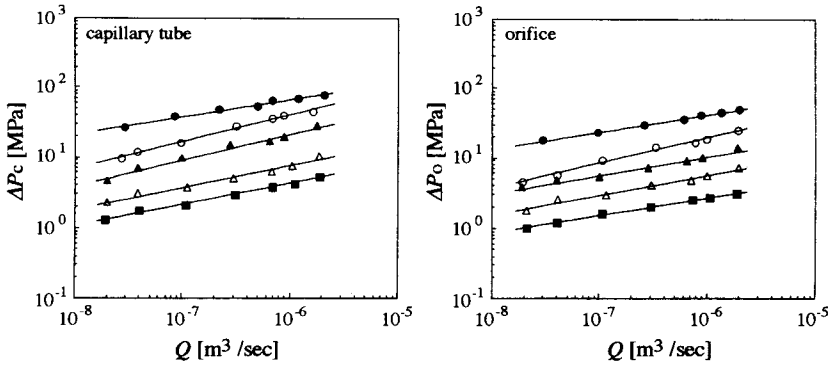Moisture content: ● 16%, ○ 20%, ▲ 25%, △ 30%, ■ 35%

Fig. 4. Relationships between flow rate and pressure drop for corn starch melts with various moisture content at 150 °C.
Moisture content: ● 16%, ○ 20%, ▲ 25%, △ 30%, ■ 35%

volumetric flow rate, Q, and obtained ΔP_d of potato and corn starch melts were shown in Fig. 5 and 6, respectively. These curves had good linearly on logarithmic scales. Apparent shear rate, $\dot{\gamma}_a$, and shear stress, τ_w, were calculated from Q and ΔP_d. The relationships between $\dot{\gamma}_a$ and τ_w of potato and corn starch melts were shown in Fig. 7 and 8, respectively. Figure 7 (A) and 8 (A) show the relationships on logarithmic scales. So the flow curves of starch melts with each moisture content had good linearly that the starch melts could be belong to power-law fluids. On the other hand, Figure 7 (B) and 8 (B) show the relationships on ordinary scales. These starch melts assumed to have some yield stress, especially at lower moisture content. From these results, the starch melts can be fitted by Herschel-Bulkley power-law model.
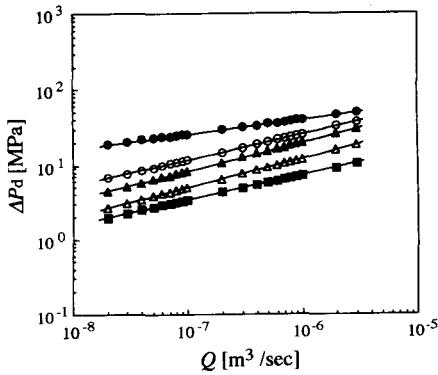
Fig. 5. Relationship between flow rate and ΔP_d for potato starch melts with various moisture content at 150 °C. Moisture content: ● 16%, ○ 20%, ▲ 25%, △ 30%, ■ 35%
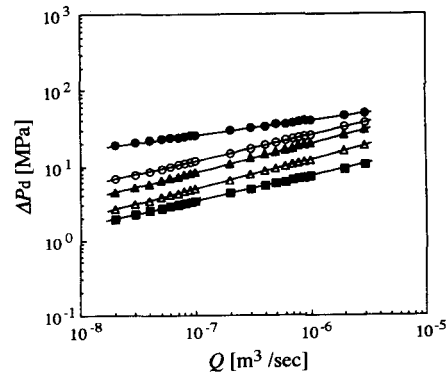
Fig. 6. Relationship between flow rate and ΔP_d for corn starch melts with various moisture content at 150 °C. Moisture content: ● 16%, ○ 20%, ▲ 25%, △ 30%, ■ 35%
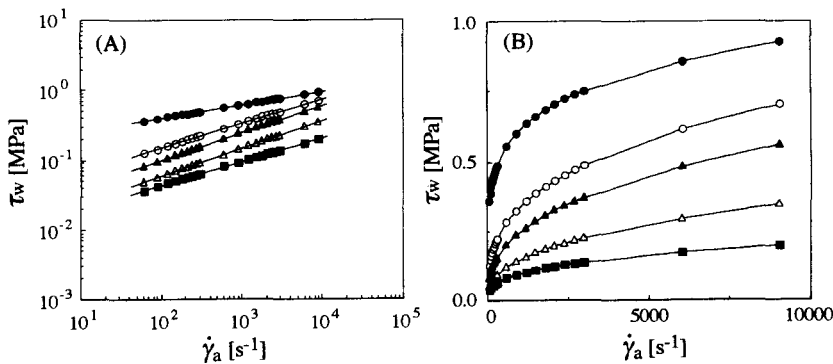

Fig. 7. Flow curves for potato starch melts with various moisture content at 150 °C on double logarithmic scale (A) and on ordinary scale (B).
Moisture content: ● 16%, ○ 20%, ▲ 25%, △ 30%, ■ 35%

Fig. 8. Flow curves for corn starch melts with various moisture content at 150 °C on double logarithmic scale (A) and on ordinary scale (B).
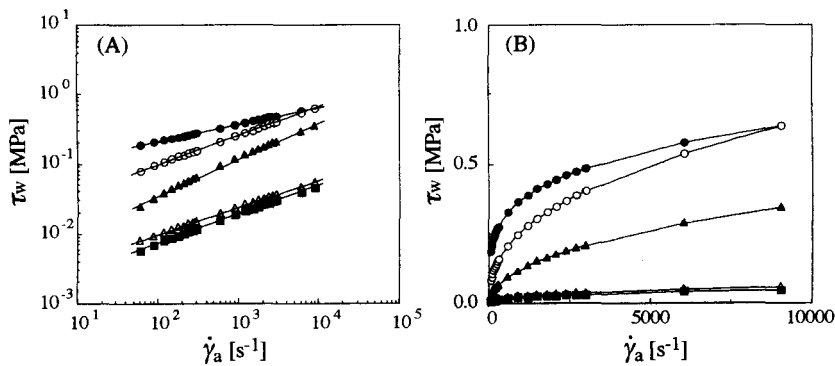Moisture content: ● 16%, ○ 20%, ▲ 25%, △ 30%, ■ 35%

Regression analysis

Figure 9 shows the relationships between moisture content and coefficients of the Herschel-Bulkley power-low model of potato starch melts. The residual sum of squares of potato starch at the moisture content of 16, 20, 25, 30 and 35% were 2.48×10^{-4}, 1.36×10^{-4}, 1.17×10^{-4}, 3.14×10^{-5} and 1.16×10^{-5}, respectively. These residual sum of squares could be small enough to fit the model to obtained data. The yield stress of potato starch melts at 16% moisture was about 0.06 MPa. Then the yield stress decreased drastically at 20% moisture and were constant value until 35% moisture. The consistency index was decreased continuously as moisture content increased. The flow behaviour index which indicate the deviation from Newtonian fluid was constant at 0.4 except 16% moisture. Figure 10 shows the relationships between moisture content and coefficients of corn starch melts. The residual sum of squares of corn starch at the moisture content of 16, 20, 25, 30 and 35% were 1.09×10^{-4}, 1.01×10^{-4}, 8.95×10^{-4}, 1.15×10^{-6} and 4.43×10^{-6}, respectively. These residual sum of squares also could be small enough to fit the model to obtained data. The yield stress of corn starch melts at 16% moisture was smaller than that of potato starch melts. The yield stress above 20% moisture and the flow behaviour
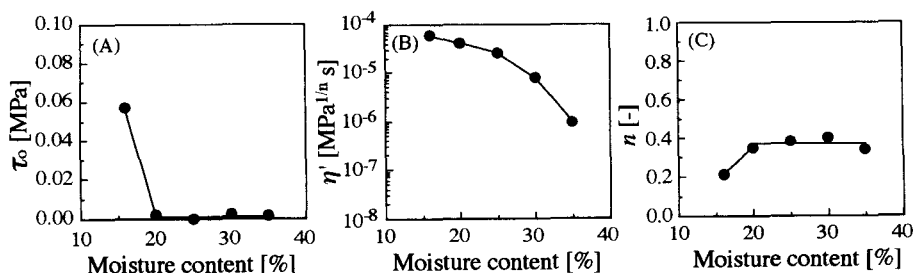

Fig. 9. Relationships between moisture content and coefficients of the Herschel-Bulkley model with potato starch.
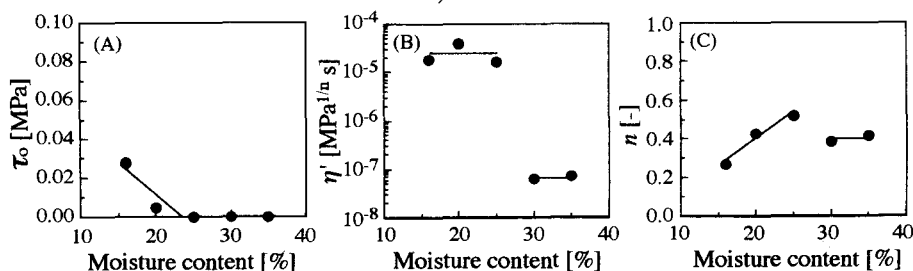(A) yield stress, (B) consistency index, (C) flow behaviour index

Fig. 10. Relationships between moisture content and coefficients of the Herschel-Bulkley model with corn starch.
(A) yield stress, (B) consistency index, (C) flow behaviour index

index of corn starch melts had similar value to those of potato starch melts. On the other hand, the consistency index of corn starch melts changed discontinuously at about 30% moisture, which was different at a point that potato starch melts changed continuously. These results suggested that the flow properties varied with moisture contents. Furthermore, the flow properties between potato and corn starch melts were different even though they were same moisture content, especially that was reflected in the consistency index. It is well known that the properties of the slurry of gelatinized starch are varied by the starch varieties. This will be effected by the size, the shape and the component of these starch particles. It was interested in the flow properties of fused starches also varied by the starch varieties.

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