

Determination of Starch Gelatinization Temperatures by Means of Polarized Light Intensity Detection

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A Brabender viscoamylograph (BV), a differential scanning calorimeter (DSC) and a polarized light detector without a microscope were used to detect potato and tapioca starches' gelatinization temperatures. Starches were ball-milled for 3 to 12 hrs to various degrees of damage. X-ray diffraction patterns indicated that potato starches were of the B-type whereas tapioca starches were of the C-type. With increasing milling time, the temperatures of the initial gelatinization of potato and tapioca starches as measured by BV, DSC and the polarized light detector were decreased. Regression analyses of the initial gelatinization temperature data of the potato and tapioca starches taken by BV and DSC, BV and the polarized light detector, and DSC and the polarized light detector measurements indicated high correlation ($R^2 > 0.95$). The analyses of initial gelatinization temperatures measured by DSC and the polarized light detector showed a linear relationships for potato and tapioca starches.

Key words: Ball milling, Brabender viscoamylograph, DSC, polarized light detector, potato, tapioca and X-ray diffraction

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งานวิจัยนี้ใช้เครื่องบราเบนเดอร์ วิสโคอิมัลโลกราฟ (BV) ดิฟเฟอเรนเชียล-สแกนนิ่ง แคลอริมิเตอร์ (DSC) และอุปกรณ์วัดปริมาณลำแสงระนาบ ตรวจวัดอุณหภูมิเจลาตินในเซชันของสตาร์ช มันฝรั่ง และสตาร์ช มันสำปะหลัง สตาร์ชจะผ่านการไม่แบบบอลลีมิลลิ่ง (3 ถึง 12 ชม.) เพื่อให้ได้ระดับความเสียหายต่างๆ บนเม็ดสตาร์ช ผลที่ตรวจสอบจาก X-ray powder diffraction พบว่าสตาร์ชมันฝรั่งเป็น B-type และสตาร์ชมันสำปะหลังเป็น C-type เวลาการไม่สตาร์ชนานขึ้น อุณหภูมิเริ่มต้นของเจลาตินในเซชันที่วัดด้วย BV DSC และอุปกรณ์ที่วัดปริมาณแสงระนาบจะลดลง การวิเคราะห์สมการแบบถดถอยแบบรีเกรสชัน ของอุณหภูมิเริ่มต้นของเจลาตินในเซชันของสตาร์ชมันฝรั่งและสตาร์ชมันสำปะหลังที่วัดจาก BV และ DSC, BV และอุปกรณ์วัดปริมาณลำแสงระนาบ และ DSC และอุปกรณ์วัดปริมาณแสงระนาบ พบความสัมพันธ์ที่สูง ($R^2 > 0.95$) ความสัมพันธ์ระหว่างอุณหภูมิเริ่มต้นของเจลาตินในเซชันของสตาร์ชทั้ง 2 ชนิด ที่วัดด้วย DSC กับอุปกรณ์วัดปริมาณแสงระนาบ เป็นลักษณะเส้นตรง

คำสำคัญ ไม่แบบบอลลีมิลลิ่ง บราเบนเดอร์ วิสโคอิมัลโลกราฟ ดิฟเฟอเรนเชียลสแกนนิ่ง-แคลอริมิเตอร์ มันฝรั่ง มันสำปะหลัง เอกซเรย์ ดิฟแฟรคชัน

INTRODUCTION

The gelatinization temperature is an important characteristic of starch properties. Many methods have been regularly used to determine the gelatinization temperatures; they are generally based on the physical changes of starch granules and suspensions during gradual heating to the full paste state. The Brabender amylograph viscometer has been used to measure the viscosity changes in starch suspensions which increase until reaching peak viscosity.⁽¹⁾ Differential scanning calorimetry (DSC) has been used to measure the enthalpy (ΔH) of starch gelatinization.⁽²⁾ The gelatinization temperature is defined as the temperature where 98% of the starch granules undergo birefringence loss when microscopically viewed with a Kofler hot stage microscope.⁽³⁾ This polarizing microscope has sufficiently high power to magnify the loss of birefringence by the small starch granules.

The purpose of this investigation was to study the feasibility of using a polarized light detector (PLD) for measuring starch gelatinization temperatures without using a microscope. The technique measured the intensity of polarized light transmitted through the starch suspension during heating. The gelatinization temperatures measured by a Brabender viscoamylograph were compared to DSC measurements. The study materials were native potato and tapioca starches ball-milled to various levels of damage in order to ensure a sufficiently wide range of gelatinization temperatures.⁽⁴⁾ A Brabender viscoamylograph, DSC and the PLD were used to determine the gelatinization temperatures of the ball-milled starches.

MATERIALS AND METHODS

Potato starch, with 17.8% moisture content, provided by Sudstarke GmbH (Germany) and tapioca starch, with 11.6% moisture content, supplied by Thai Wah Co., Ltd. (Thailand) were used as the starch materials.

Ball-mill treatment

Ceramic balls (36 balls in 3 different diameters: 1.5 cm, 2 cm and 3 cm; 12 balls of each diameter) with a total weight of 660 g and starch (100 g) were put in a ceramic ball-mill container (rolling-type, 1 kg capacity cylindrical container). Ball-milling speed was set at 138 rpm at ambient temperature for

various milling durations (3, 6, 9 and 12 hrs). Untreated starches were taken as the control samples and all millings were done in triplicate. After milling, each starch was kept in a sealed aluminum foil bag until used.

An enzyme kit from Megazyme Co. was used to assess the damaged starches spectrophotometrically.⁽⁵⁾

Brabender Viscoamylograph

A Brabender viscoamylograph (model Viscograph PT 100) with a 700 cmg cartridge was used to study the gelatinization process of potato starch at 4.0% concentration (dry weight basis, dwb) and tapioca starch at 6% dwb.⁽¹⁾ The starch was dissolved in distilled water in a sample cup to give a total weight of 460 gm. Samples were heated at a rate of 1.5°C/min from room temperature to 95°C and maintained at 95°C for 20 minutes. The gelatinization temperature range was defined as being from the initial pasting temperature (T_i) to the peak viscosity temperature (T_f). All sample preparations were repeated in triplicate.

DSC Method

The differential scanning calorimetry (DSC) used was a Perkin-Elmer DSC - 7. The starch sample (3 mg) was weighed in an aluminum pan and 6 μ l of water was added. The pan was then hermetically sealed using a sample sealing accessory (Perkin - Elmer 0219-0061) and equilibrated overnight at room temperature, before being scanned at a heating rate of 10°C/min with an empty pan as the reference. The starch thermal transitions were defined in terms of T_o (onset), T_p (peak) and T_c (conclusion of gelatinization temperature).⁽²⁾ The enthalpy (ΔH , J/g) of the DSC endotherm was calculated. All sample preparations were repeated in triplicate.

The polarized light detector

Figure 1 shows the systems main parts which consisted of a light-emitting diode (LED) as light source, a light-dependent resistor (LDR) as the light detector and a semiconductor temperature sensor to detect the temperature of the starch suspension. The system with the above mentioned devices and also a heating element was used to measure the starch granule birefringence loss by

observing the polarized light intensity decrease during the heating of the starch. A number of resistors were used as heating elements and a pair of polaroids served as the polarizer and the analyzer. An electronic circuit served to perform the signal processing, controlling and measuring of the gelatinization temperature.

To determine the starch birefringence loss, the polaroids were initially set for extinction by orientating the analyzer transmission axis (TA) at 90° relative to the

TA of the polarizer. A glass slide containing $50 \mu\text{l}$ of aqueous starch suspension (1% dwb) was placed on the heating elements between the analyzer and the polarizer. The E-vector of the light rotated by the starch sample eliminated the condition of extinction. The transmitted light intensity varied according to the birefringence property and was measured by the LDR detector. A graph of light intensity versus temperature was developed for each experiment. Heating was at the rate of $1.5^\circ\text{C}/\text{min}$. All sample preparations were repeated in triplicate.

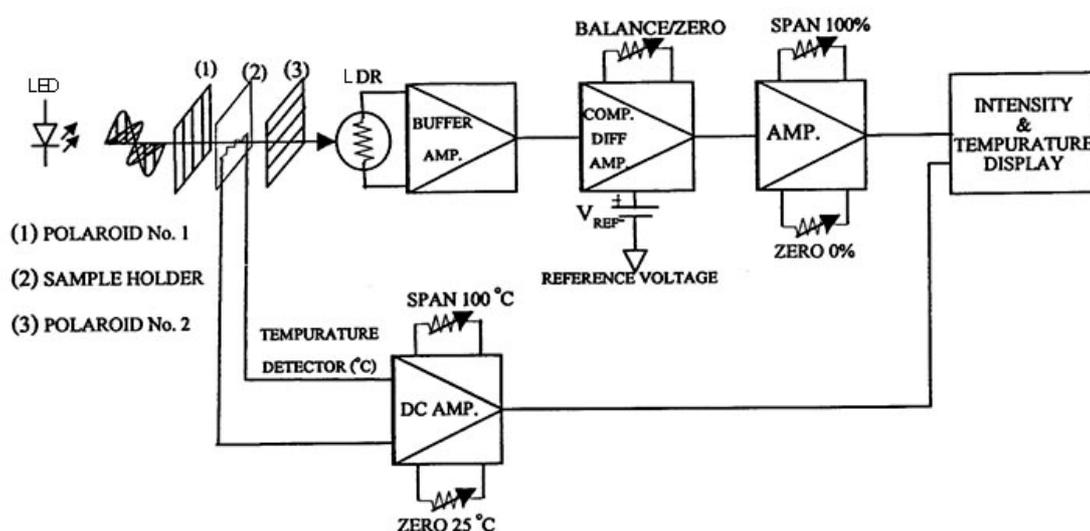


Figure 1. Schematic diagram of the equipment.

Statistical analysis

All data from each experiment were analyzed for variance. Means of all replications were used to calculate least significant differences. Relationships for each instrumental method were analyzed by regression analysis.

RESULTS AND DISCUSSION

Ball-milling significantly increased the damage of the potato and tapioca starches (Table 1). The percentage of damaged potato starch was higher than tapioca starch. The larger potato starch granule surface receiving the impact force might be the cause of the higher degree of damage.⁽⁴⁾ The size of the potato and tapioca granules are in the range of $5\text{-}100 \mu$ (mean 30μ) and $4\text{-}35 \mu$ (mean 20μ), respectively.⁽⁶⁾

Table 1. Percentage of damaged potato and tapioca starch granules from ball-milled treatment.

Milling [hr]	% damaged granules ^(*)	
	Potato	Tapioca
0	0.60 ^e	0.62 ^e
3	7.90 ^c	3.13 ^d
6	12.46 ^a	4.85 ^a
9	15.08 ^b	8.88 ^b
12	20.08 ^a	12.54 ^a

^{a,b,c} Means with different superscripts within a column differ significantly at $p \leq 0.05$

The X-ray diffraction patterns of potato and tapioca starches after ball-milling for 12 hours are shown in Figure 2. The potato starches were of the B-type whereas tapioca starches were of the C-type. The peak d-spacings of the B-type pattern were around 15.8, 5.16, 4.0 and 3.70 Å, while those of the C-type pattern were 5.78, 5.12, 4.85 and 3.78 Å.⁽⁷⁾ The peak intensities decreased with increasing milling time. Ball-milling wheat and maize starches has been found to increase the low molecular weight amylopectin content.⁽⁴⁾ It was reported that the composition

and physical properties of the amorphous damaged starch were different from the native starch granules. The crystallinity of starch granules was negatively correlated ($r = -0.992$, $n = 7$) to the damaged starch content,⁽⁸⁾ the regression gave zero crystallinity at 94.3% damaged starch. After ball-milling for 12 hrs, the percentage of damaged potato and tapioca starch averaged 20 and 13 respectively and therefore, their crystallinity was mostly retained. It takes over 80 hrs of ball-milling treatment in order to fully gelatinize potato starch.⁽⁹⁾

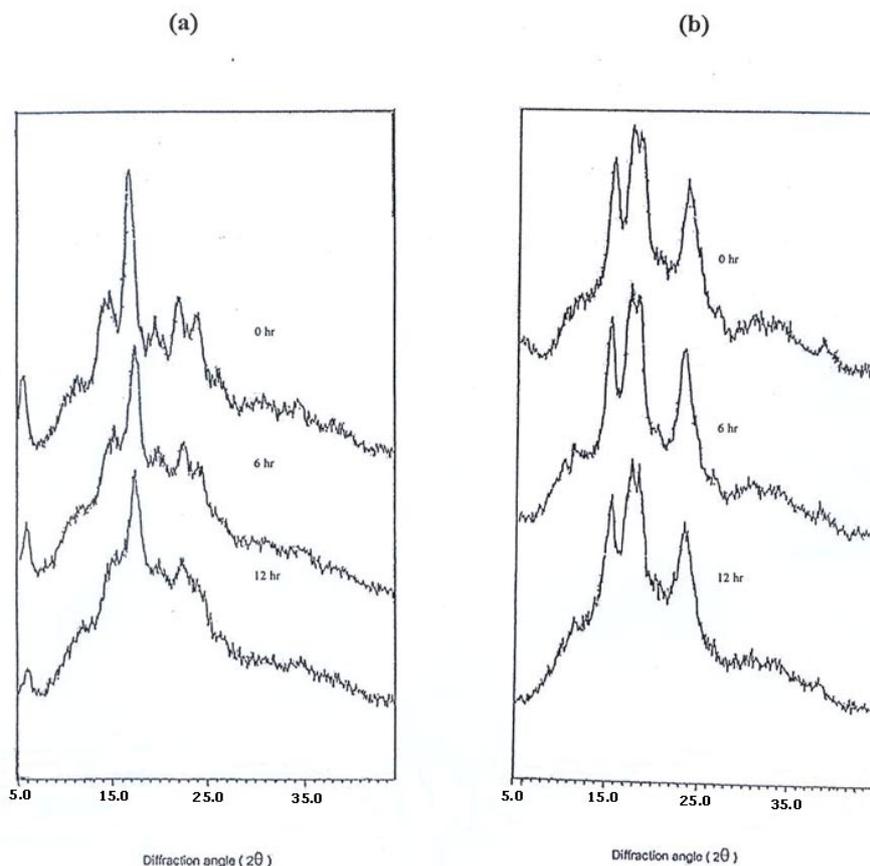


Figure 2. X-ray diffraction pattern of (a) potato starch and (b) tapioca starch after various ball-milling times.

The enthalpies of gelatinization (ΔH) of native potato and tapioca are 18.8 J/g and 16.7 J/g respectively.⁽¹⁰⁾ When measured by DSC, the enthalpies (ΔH) of our native potato and tapioca samples were similar to the above. The gelatinization enthalpies of our native and ball-milled potato and tapioca samples are shown in Table 2. It is known that the ΔH of damaged starches declined with increased milling time.⁽⁴⁾ Decreases in T_o , T_p , T_c and ΔH of damaged starches with increasing milling time were observed in both starches. For potato, T_p changed from 63.4°C at 0 hr

down to 61.7°C after 6 hrs and 60.1°C after 12 hrs., and ΔH significantly decreased by 3.8 J/g after 3 hrs of ball-milling and gradually decreased a further 3.9 J/g after an additional 9 hrs of milling. For tapioca, after 12 hrs of ball-milling, the enthalpy decreased by 3.6 J/g. The gradual decrease in enthalpy with increasing milling time is confirmed and supported by the findings of Tamaki *et al.* (1997), that gelatinization spreads stepwisely from the surface towards the inner part of the granules.

Table 2. DSC properties of ball-mill treated potato and tapioca starch.

a. Potato starch

Milling time (hr)	Gelatinization temperature* (°C)			ΔH (J/gm)
	T_o	T_p	T_c	
0	59.56 ^a	63.36 ^a	75.73 ^a	19.67 ^a
3	56.24 ^b	62.81 ^b	73.92 ^b	15.91 ^b
6	54.48 ^c	61.68 ^c	73.69 ^b	14.10 ^c
9	54.50 ^c	61.45 ^c	73.95 ^b	13.64 ^c
12	53.57 ^d	60.14 ^d	73.32 ^b	11.98 ^d

b. Tapioca starch

Milling time (hr)	Gelatinization temperature* (°C)			ΔH (J/gm)
	T_o	T_p	T_c	
0	61.89 ^a	67.60 ^a	81.44 ^a	15.97 ^a
3	59.59 ^b	66.39 ^b	79.93 ^b	15.24 ^{ab}
6	57.65 ^c	66.09 ^b	79.73 ^b	14.60 ^{bc}
9	54.49 ^d	65.42 ^c	79.30 ^b	13.93 ^c
12	52.32 ^e	65.49 ^c	79.31 ^b	12.35 ^d

* T_o (onset temperature), T_p (peak temperature), T_c (conclusion temperature), ΔH = enthalpy of gelatinization

^{a,b,c} Means with different superscripts within a column differ significantly at $p \leq 0.05$

Gelatinization temperatures determined by the Brabender amylograph are shown in Table 3. The initial gelatinization temperatures of both starches decreased as the milling time increased. Milling had a more significant affect on potato starch than tapioca. The results showed that on increasing the milling

time, the starch granules would hydrate and swell to give a viscosity of 10 BU faster than the native starch granules. It is known that physical damage converts the large ordered regions into disorderly amorphous materials, freely accessible to water.⁽⁴⁾

Table 3. Gelatinization temperature range of potato and tapioca starches measured with a Barbender viscograph.

Starch	Gelatinization temperature (°C)*	Milling time (hr)				
		0	3	6	9	12
Potato	T _i	65.1 ^a	63.9 ^b	62.9 ^c	62.1 ^d	60.5 ^e
	T _f	85.8 ^{ab}	87.0 ^a	87.0 ^a	86.6 ^a	84.1 ^b
Tapioca	T _i	67.6 ^a	66.9 ^b	66.1 ^c	65.4 ^d	64.4 ^e
	T _f ^{ns}	76.0	77.0	76.2	76.5	76.5

* T_i = initial temperature (°C) when the viscosity was 10 BU (Brabender Units), T_f = peak viscosity temperature (°C)

^{a,b,c}Means with different superscripts within a row differ significantly at p ≤ 0.05

The transmitting percentage of polarized light intensity and temperature of native potato and tapioca starches at heating rate of 1.5°C/min, as determined by the polarized light detector are shown in Table 4. The ranges of the potato and tapioca gelatinization temperatures were between 58.6 and 65.5°C (Δ6.9°C), and between 60.8 and 65.0°C (Δ4.2°C), respectively.⁽¹¹⁾ The ranges of potato and Siamese tapioca starches gelatinization temperatures measured by using a Kofler microscopic technique were between 50 and 68°C, and between 62 and 73°C,

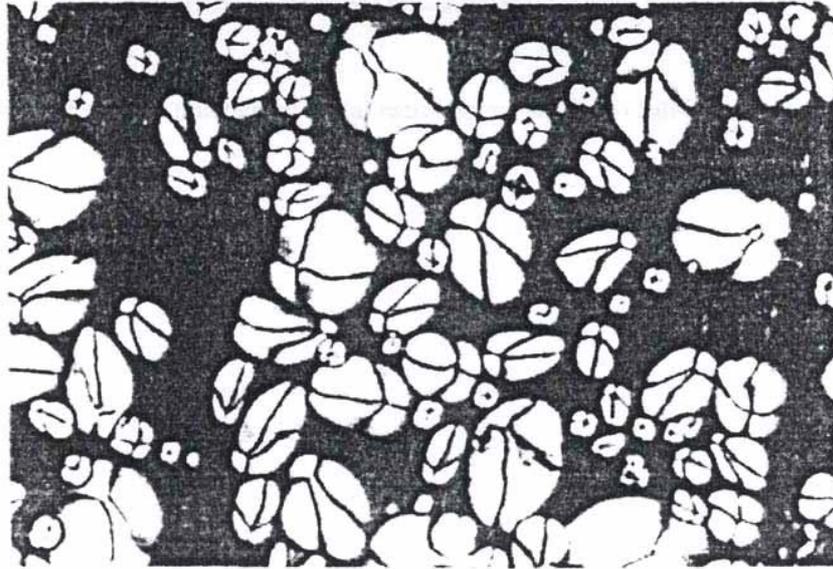
respectively. Several studies have been done on the ranges of tapioca starch gelatinization temperatures measured by using microscopic techniques, for example, 57.0-65.0°C,⁽¹²⁾ 58.5-70°C⁽¹³⁾ and 58.5-68.0°C.⁽¹⁴⁾ The ranges of the gelatinization temperatures measured by the polarized light detector were narrower than those reported, probably resulting from the thermal diodes being attached to the microscopic slide (starch holding cells) and, therefore, measuring the gelatinization temperatures of the starch suspension within the cell.

Table 4. Percentage polarized light intensity during the gelatinization of native potato and tapioca starches, as measured with the polarized light detector.

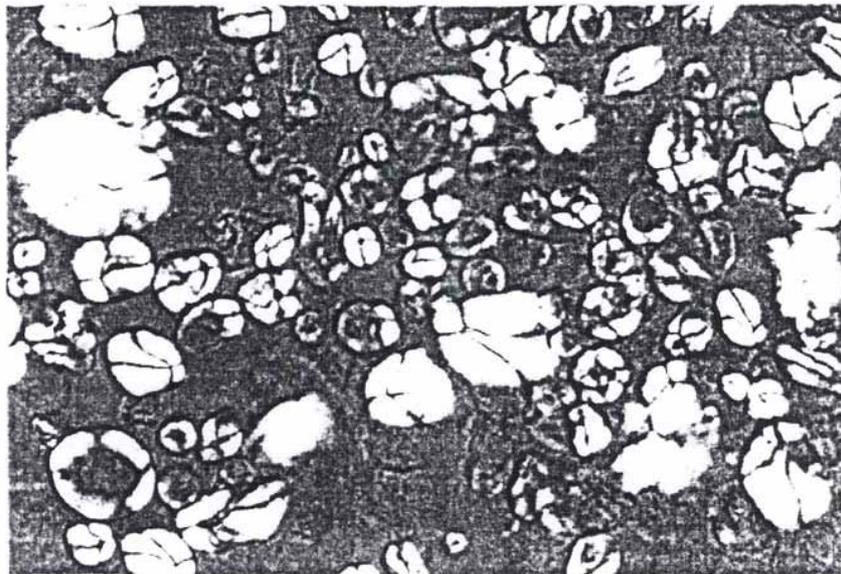
%Polarized light intensity	Gelatinization temperature (°C) ^a			
	Potato		Tapioca	
	Mean±SD	%CV	Mean±SD	%CV
98	58.6±1.1	1.88	60.8±0.8	1.32
95	59.4±0.9	1.52	61.4±0.7	1.14
90	60.2±0.9	1.50	62.0±0.5	0.81
80	61.1±0.8	1.31	62.9±0.4	0.64
70	61.7±0.7	1.13	64.0±0.5	0.78
60	62.1±0.7	1.13	65.0±0.5	0.77
50	62.7±0.6	0.96		
40	63.4±0.7	1.10		
30	64.2±0.7	1.09		
20	65.5±0.6	0.92		

CV is the coefficient of variation

SD is standard deviation



(a)



(b)

Figure 3. Polarized micrograph of (a) native potato starch (b) ball-milled potato starch.

Figure 3 shows the polarized light micrograph of native and ball-milled starch samples. The temperature at which the initial polarized light intensity of a starch sample reached 98% was recorded and when the light intensity declined to a constant value, the ending point was taken. The tapioca's end reading was at 60% of light intensity, higher

than the potato's 20% because tapioca starch granules are smaller than those of potato. In order to detect the gelatinization temperature of tapioca below the 60% polarized light intensity, the light signal must be amplified. Table 5 shows the means and standard deviations of the gelatinization temperatures of native and ball-mill treatment potato and

tapioca starches. As milling time increased, the initial gelatinization temperatures (at 98% light intensity) of the starches decreased. This tendency agreed with the DSC results. The damage caused loss of birefringence as shown by the polarized light detector.

The initial gelatinization temperatures from the Brabender amylograph (Table 3) were higher than the onset temperatures (T_o) from DSC (Table 2) and the temperatures

measured by the polarized light detector (Table 5). The Brabender amylograph measures the temperature of gelatinizing at the initial detectable viscosity (10 BU) of the starch suspension, while DSC indicates the crystalline structure melting of the starch granules and the loss of birefringence which may proceed before any significant swelling and hydration of starch granules.⁽¹⁰⁾

Table 5. Gelatinization temperatures of ball-mill treated potato and tapioca starches measured with the polarized light detector.

a) potato starch

%Polarized light intensity	Gelatinization temperature*(°C) after different milling times (hr)				
	0	3	6	9	12
98	58.6 ^a	51.7 ^b	50.2 ^{bc}	49.1 ^{cd}	48.1 ^d
95	59.4 ^a	53.9 ^b	52.5 ^{bc}	51.3 ^{cd}	50.8 ^d
90	60.2 ^a	55.8 ^b	54.4 ^c	53.1 ^{cd}	52.9 ^d
80	61.1 ^a	57.7 ^b	56.5 ^{bc}	55.3 ^{cd}	55.0 ^d
70	61.7 ^a	58.7 ^b	57.7 ^{bc}	56.5 ^c	56.6 ^c
60	62.1 ^a	59.5 ^b	58.7 ^{bc}	57.7 ^d	57.8 ^{cd}
50	62.7 ^a	60.3 ^b	59.5 ^{bc}	58.4 ^d	58.8 ^{cd}
40	63.3 ^a	61.1 ^b	60.3 ^{bc}	59.1 ^d	59.7 ^{cd}
30	64.1 ^a	61.9 ^b	61.2 ^{bc}	59.9 ^d	60.6 ^{cd}

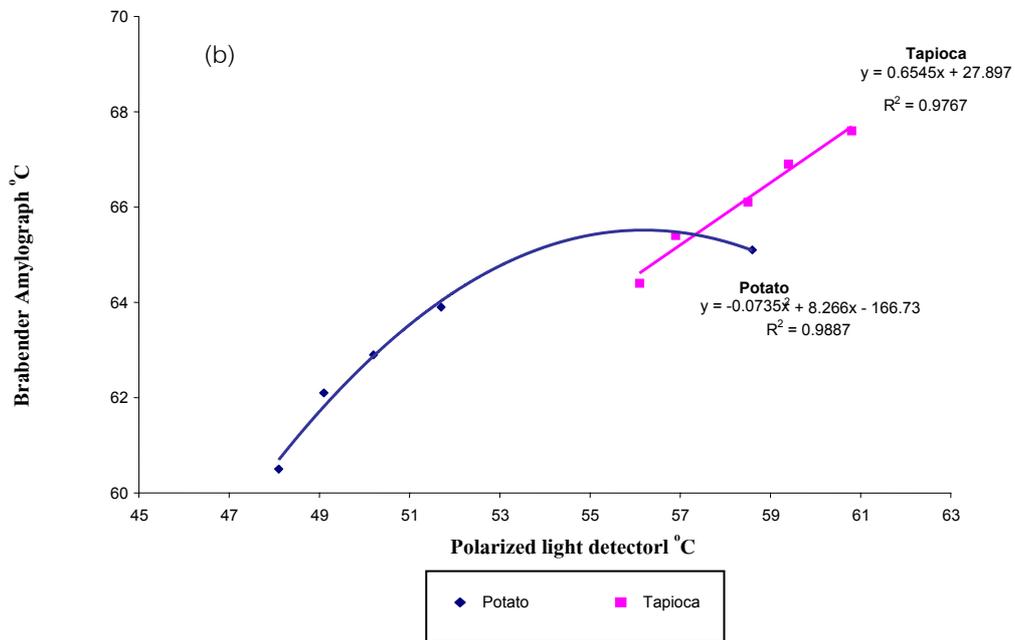
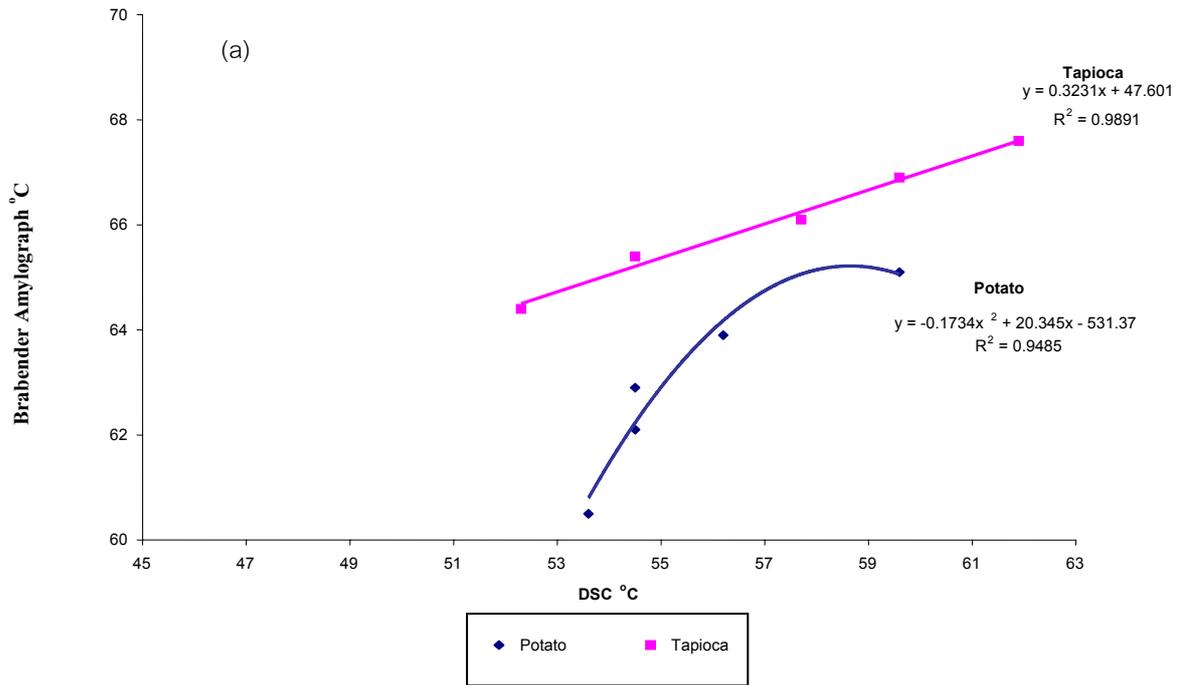
b) tapioca starch

%Polarized light intensity	Gelatinization temperature*(°C) after different milling times (hr)				
	0	3	6	9	12
98	60.8 ^a	59.4 ^{ab}	58.5 ^{bc}	56.9 ^d	56.1 ^d
95	61.4 ^a	60.1 ^{ab}	59.3 ^{bc}	58.0 ^d	57.2 ^d
90	62.0 ^a	60.9 ^{ab}	60.1 ^{bc}	59.1 ^{cd}	58.5 ^d
80	63.0 ^a	62.2 ^{ab}	61.4 ^{bc}	60.7 ^{cd}	60.2 ^d
70	63.9 ^a	63.3 ^{ab}	62.5 ^{bc}	61.9 ^{cd}	61.5 ^d
60	65.0 ^a	64.4 ^{ab}	63.6 ^{bc}	63.1 ^c	62.8 ^c

^{a,b,c}Means with different superscripts within a row differ significantly at $p \leq 0.05$

The regression analyses of the initial gelatinization temperatures of the starches as measured by the three instruments are shown in Figure 4. The results indicated high correlation ($R^2 > 0.95$) for the initial gelatinization temperatures between DSC and the polarized light detector, Brabender amylograph and the polarized light detector and between the Brabender amylograph and DSC. For the DSC and the polarized light detector, linear relationships were observed for the initial and final gelatinization temperatures of the potato and tapioca

starches [Figure 4c and 5]. The relationship between the measurement of the Brabender amylograph and the DSC, and Brabender and the polarized light detector, (Figure 4a and 4b) gave second order polynomial responses ($R^2 = 0.95-0.99$) for potato starch while those for tapioca starch gave linear responses ($R^2 = 0.98-0.99$). These features may relate to the large granules of potato starch (5-100 μm , diameter)⁽⁶⁾ and also the difference of the ordered crystalline structure of the starch granules.⁽¹⁵⁾



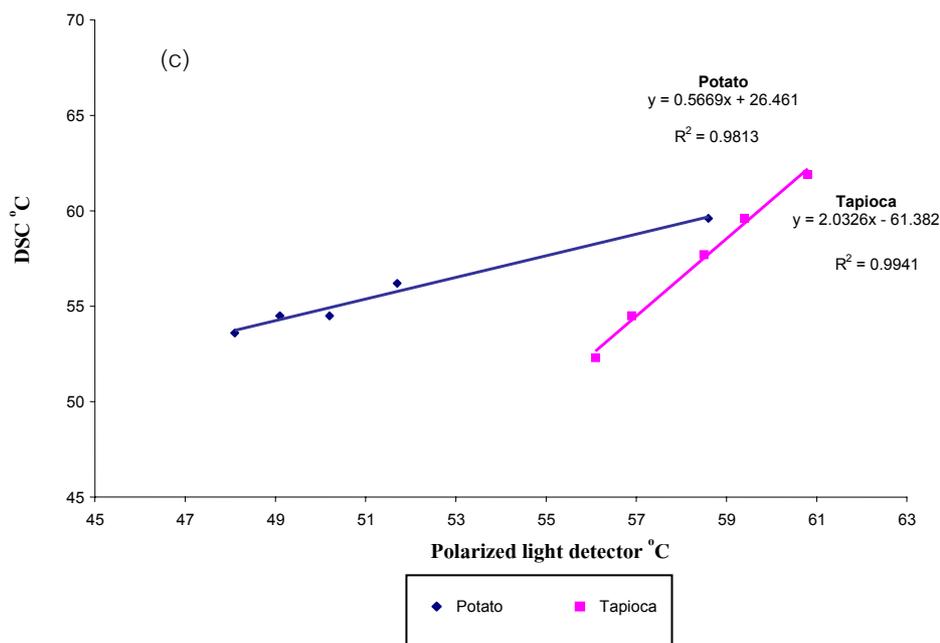


Figure 4. Regression analysis of initial gelatinization temperature between (a) Brabender amylograph and DSC (b) Brabender amylograph and the polarized light detector (c) DSC and polarized light detector.

CONCLUSIONS

The least time consuming method among the three instruments used to measure starch gelatinization temperature is the polarized light detector. It is characterized by a temperature reading accuracy of 0.1°C, and shows high reproducibility. The polarized light detection technique may provide a mean for rapid screening of starch initial gelatinization temperatures. The technique has been proved with high correlation to DSC. However, other experiments are still required to explain the pasting characteristic of starch.

REFERENCES:

1. Thiewes, H. J. and Steeneken, P. A. M. (1997) "Comparison of the Brabender Viscograph and the Rapid Visco Analyzer. I. Statistical evaluation of the pasting profile" *Starch / Starke*. **49**, 85-92.
2. Young, S. K., Wiesenborn, D. P., Orrand, P. H. and Grant, L. A. (1995) "Screening potato starch for novel properties using differential scanning calorimetry" *Journal of Food Science* **60**, 1060-1065.
3. French, D. (1984) "Organization of starch granules" *Starch : Chemistry and Technology*. (edited by R. L. Whistler, J. N. BeMiller & E. F. Paschall). New York, Academic Press, 234.
4. Morrison, W. R., Tester, R. F. and Gidley, M. J. (1994) "Properties of damaged starch granules. II Crystallinity, molecular order and gelatinization of ball-milled starches" *Journal of Cereal Science* **19**, 209-217.
5. AACC, (1995) *Approved Methods of the AACC*. St. Paul, MN : American Association of Cereal Chemists.
6. Ellis, R. P., Cochrane, M. P., Dale, M. F. B., Duffus, C. M. Lynn, A., Morrison, I. M., Prentice, R. D. M., Swanston, J. S. and Tiller, S. (1998) "Starch Production and Industrial Use" *Journal of the Science of Food and Agriculture*. **77**, 289-311.
7. Zobel, H. F. (1964) "X-ray analysis of granule starches" *Methods in Carbohydrate Chemistry. IV*. (edited by R. L. Whistler, R. J. Smith, J. N. BeMiller and M. L. Wolform). New York, Academic Press. 109-113.

8. LeLievre, J. (1974) "Starch damage" *Starch/Starke* **26**, 85-88.
9. Tamaki, S., Hisamatsu, M., Teranishi, K. and Yamada, T. (1997) "Structural change of potato starch granules by ball-mill treatment" *Starch/Starke* **49**, 431-438.
10. Zobel, H. F. (1984) "Gelatinization of starch and mechanical properties of starch pastes" *Starch: Chemistry and Technology*. (edited by R. L. Whistler, J. N. BeMiller & E.F. Paschall) New York: Academic Press, 291-293.
11. Snyder, E. M. (1984) "Industrial microscopy of starches" *Starch : Chemistry and Technology* (edited by R. L. Whistler, J. N. BeMiller & E. F. Paschall): New York : Academic Press, 661-673.
12. Rasper, V., Rasper J and Mabey G. L. (1974) "Functional properties of non-wheat flour substitutes in composite flours I" *Journal of the Canadian Institute of Food Science & Technology* **7**, 86-97.
13. Moorthy, S. N. (1985) "Acetylation of cassava starch using perchloric acid catalysis" *Starch/Starke* **37**, 307-308.
14. Franco, C. M., Ciacco C. F. and Tavares D. Q. (1998) "Studies on the susceptibility of granular cassava and corn starches to enzymatic attack. Part 2. Studies of the granular structure of starch" *Starch /Starke* **40**, 29-32.
15. Yuan, R. C., Thompson, D. B. and Boyer, C. D. (1993) "Fine structure of amylopectin in relation to gelatinization and retrogradation behavior of maize starches from three wx-containing genotypes in two inbred lines" *Cereal Chemistry* **70**, 81-89.

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