Relationship between Intrinsic Viscosity, Thermal, and Retrogradation Properties of Amylose and Amylopectin

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Abstract

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The relationships between intrinsic viscosity and some properties of amylose and amylopectin were investigated. The intrinsic viscosities determined by Ubbelohde viscometer for rice, maize, wrinkled pea and potato amyloses were 46.28 ± 0.30, 123.94 ± 0.62, 136.82 ± 0.70, and 167.00 ± 1.10 ml/g, respectively; and the intrinsic viscosities of rice, maize, wrinkled pea and potato amylopectins were 77.28 ± 0.90, 154.50 ± 1.10, 162.56 ± 1.20 and 178.00 ± 1.00 ml/g, respectively. The thermal and retrogradation properties of amylose and amylopectin were investigated by differential scanning calorimeter (DSC). Results showed that the thermal enthalpy (ΔH_g) was positively correlated with intrinsic viscosity, however, the onset and peak temperatures were not related to the intrinsic viscosity. The amylose and amylopectin retrogradation enthalpy values were negatively related to intrinsic viscosity, while the onset and peak temperature values of retrograded amylose and amylopectin were not related to the intrinsic viscosity during storage (except one-day storage). Furthermore, the onset and peak temperatures and retrogradation enthalpy of amylose and amylopectin changed slowly during storage at 4°C.

Keywords: DSC; enthalpy; storage; corn starch

Starch is made up of amylose and amylopectin; amylose and amylopectin are two important components of starchy foods, the properties of amylose and amylopectin are related to the eating quality of starchy foods. Therefore, it is important to study the properties of amylose and amylopectin in order to produce high-quality starchy foods.

Recently, many researchers have reported various properties of amylose such as structural conformation and stability in solvents (Nakanishi *et al.* 1993; Shimada *et al.* 2000; Radosta *et al.* 2001; Tusch *et al.* 2011), conformational transitions (Cheetham & Tao 1997), crystallisation and crystal structure (Takahashi *et al.* 2004; Creek *et al.* 2006; Popov *et al.* 2009; Montesanti *et al.* 2010), amylose complexes (Hulleman *et al.* 1996; Ozcan & Jackson 2002; Nuessli *et al.* 2003; Ciesielski & Tomasik 2004; Cardoso *et al.* 2007; Nishiyama *et al.* 2010), gel microstructure and textural properties (Torres *et al.* 1978; Leloup *et al.* 1992) and amylose gel physical network (Lay & Delmas 1998). Whereas, research on the properties of amylopectin was focused on its structural and retrogradation properties (Manners & Matheson 1981; Manners 1989; Paredes-Lopez *et al.* 1994), crystallinity of amylopectin films and network formation (Putaux *et al.* 2000; Myllarinen *et al.* 2002), and amylopectin complexes (Ciesielski & Tomasik 2004). However, little information is available about the intrinsic viscosity, thermal and retrogradation properties of amylose and amylopectin from different starches. The relationship between the intrinsic viscosity, thermal and retrogradation properties of amylose and amylopectin is still not clear. Therefore, the objective of the present study was

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to investigate thermal and retrogradation properties of amylose and amylopectin, and their relationship with intrinsic viscosity of amylose and amylopectin from different starches.

MATERIAL AND METHODS

Materials

Starch. Rice and pea starch was isolated from Japonica rice (Northeastern Rice, Qiqihar city, Heilongjiang province, China) and wrinkled pea (Qiqihar city, Heilongjiang province, China) by using alkaline steeping methods reported by Wang *et al*. (2011) and Yu *et al*. (2012a), respectively. Maize starch was purchased from the Heilongjiang Jingpo Lake Agricultural Development Co., Ltd. (Mudanjiang city, Heilongjiang province, China), and potato starch was purchased from the Nehe Snow Co. Ltd. (Nehe city, Heilongjiang province, China). The starch obtained from different sources was dried separately at 40°C for 48 h, passed through a 100-mesh sieve (0.15 mm) and stored at 4°C. All other chemicals used were of analytical grade unless otherwise stated.

Amylose and amylopectin preparation. Amylose and amylopectin were extracted from different kinds of starches by earlier reported methods with some modifications (Zhong *et al*. 2006; Chen *et al*. 2008; Yu *et al*. 2012b). Starch (10 g dry basis – DB) was soaked with 10 ml of ethanol and 350 ml of 0.5 mol/l NaOH, and dissolved by placing in boiling water with stirring for 30 min, then the solution was centrifuged (5000 *g*, 10 min) and the supernatant was collected. The process was repeated for the second cycle, and both supernatant fractions (first and second) were combined, and to this 200 ml of 1-butanol and isoamyl alcohol (3 : 1, v/v) was added. The mixture was stirred and held at boiling temperature for 10 min, then cooled to room temperature. The mixture was held at 4 ± 1°C in a refrigerator for 24 h, and centrifuged (10 000 *g*, 15 min, 4°C). The precipitate was used to isolate amylose, while the supernatant was used for amylopectin isolation.

Amylose preparation: Precipitate was suspended in aqueous 10% 1-butanol (one-third of the volume of supernatant), and the suspension was boiled under stirring for 1 h, cooled and stored at 4°C for 24 h, and centrifuged. All the steps were repeated five times to purify amylose. Finally, the amylose-1-butanol complex was collected by centrifugation, washed with ethanol at least twice, and freeze-dried. The dried powder was passed through a 100-mesh sieve (0.15 mm) and stored at 4°C until further use.

Amylopectin preparation: The supernatant was mixed with 400 ml of ethanol, incubated at room temperature for 24 h, and centrifuged. All the steps were repeated five times to purify amylopectin, and the fractionated amylopectin was freeze-dried, then passed through a 100-mesh sieve and stored at 4°C until further use.

Intrinsic viscosity

Preparation of solutions. Solutions were prepared according to the method of Everett and Foster (1959) with some modifications. Samples (0.4 g DB) of amylose or amylopectin were dispersed in dimethyl sulfoxide in a beaker with stirring, and transferred to a 100 ml volumetric flask to have the mass ratio of 0.4% (w/v). The solution or solvent was clarified for viscometric measurements by passing it rapidly through a medium-sized glass filter.

Determination of intrinsic viscosity. The intrinsic viscosity was determined according to earlier reported methods with some modifications (Everett & Foster 1959; Cowie 1960; Leach 1963). The flow time was determined by Ubbelohde viscometer with a capillary internal diameter of 0.57 mm and the determining ball volume of 4.0 ml (Shanghai Shengli Glass Instrument Co., Ltd., Shanghai, China). All the measurements were carried out in a water bath at 25.0 ± 0.1°C, the viscosities in dimethyl sulfoxide were determined in Ubbelohde viscometer (Shanghai bodi Chemical & Technology Co., Ltd., Shanghai, China) which had a flow time for the solvent of about 241 seconds. For determination of flow time in Ubbelohde viscometer, 10 ml of solvent or solution was added to the viscometer and was equilibrated in a water bath at a constant temperature for 10 minutes. The flow time was determined by a stopwatch for several concentrations of a given sample, the viscosity numbers and $(\eta - \eta_0)/\eta_0 C$; were: η – viscosity of solution; η_0 – viscosity of the solvent; C – concentration of solution. The limiting viscosity numbers were determined by plotting $(\eta - \eta_0)/\eta_0 C$ vs. C and extrapolating to the zero concentration. All the measurements were performed in triplicates and the results are presented as mean values ± standard deviation.

Differential scanning calorimetry

Thermal and retrogradation properties of samples were analysed by a differential scanning calorimeter (DSC Q20; TA Instruments, New Castle, USA). A total weight of 3.0 mg samples (dry basis) and distilled water (1 : 2, w/w) was placed in pre-weighted aluminium sample pans (TA060601), and then sealed hermetically to prevent moisture loss and kept overnight. For all DSC runs, a sealed empty aluminium pan was used as reference. The sample was held isothermally at 20°C for 1 min before heating from 20 to 150°C at 10°C/minute. The onset, peak and end temperature, and gelatinization enthalpy (ΔH_g, J/g) (amylose ranged from 110~140°C; amylopectin from 40~100°C) were calculated. Then, the pans were stored at 4°C for 14 days, and were heated again. The onset, peak temperature and enthalpy (ΔH_r, J/g) associated with the retrograded amylose (120~140°C) or retrograded amylopectin (40~70°C) were calculated. The DSC measurements were performed in triplicate. All the results are presented as mean values ± standard deviation.

Statistical analysis. One-way analysis of variance (ANOVA) and Duncan's multiple-range test were performed by the procedure of SAS 8.0 (SAS Institute, Cary, USA).

RESULTS AND DISCUSSION

Intrinsic viscosity

The intrinsic viscosities of amylose and amylopectin were determined. As shown in Table 1, the intrinsic viscosities of rice, maize, wrinkled pea, and potato amylose were 46.28 ± 0.30, 123.94 ± 0.62, 136.82 ± 0.70, and 167.00 ± 1.10 ml/g, respectively. In general, intrinsic viscosity of amylose depends on molecular weight (Foster & Hixon 1944; Cowie 1961; Ahmad *et al.*1999; Xu *et al.* 2007), and increases with molecular weight (Mua & Jackson 1997). From these results, we can conclude that the molecular weight of potato amylose was the highest, and that of rice amylose was the lowest. This point of view is in accordance with the results of earlier reports (Roger & Colonna 1996; Mua & Jackson 1997). The intrinsic viscosity value of rice amylose was similar to the report of Xu *et al.* (2007). However, the intrinsic viscosity values of maize amylose and rice amylose were lower than the reported results (Roger & Colonna 1996; Ahmad *et al.* 1999), which may have been caused by different solvents.

On the other hand, the intrinsic viscosities of rice, maize, wrinkled pea and potato amylopectin (Table 1) were 77.28 ± 0.90, 154.50 ± 1.10, 162.56 ± 1.20, and 178.00 ± 1.00 ml/g, respectively. Intrinsic viscosity of rice amylopectin was the lowest, and that of potato amylopectin was the highest. The molecular shape and conformation can significantly influence the intrinsic viscosity of amylopectin in solution (Durrani & Donald 2000). The intrinsic viscosities of amylopectin were higher than those of amylose, which may be due to their differences in molecular weight and structure. The amylose and amylopectin structures were different in different solutions (Putaux *et al.* 2000; Zhong *et al.* 2006), and the molecular shape and conformation are also related to intrinsic viscosity (Cowie 1960; Ahmad *et al.* 1999).

Table 1. Intrinsic viscosity of amylose and amylopectin from different starches

Starch	Intrinsic viscosity (ml/g)	
	amylose	amylopectin
Rice	$46.28 \pm 0.30^{d,B}$	$77.28 \pm 0.90^{d,A}$
Corn	$123.94 \pm 0.62^{c,B}$	$154.50 \pm 1.10^{c,A}$
Wrinkled pea	$136.82 \pm 0.70^{b,B}$	$162.56 \pm 1.20^{b,A}$
Potato	$167.00 \pm 1.10^{a,B}$	$178.00 \pm 1.00^{a,A}$

^{A,B}numbers followed by the same uppercase superscript letters in the same row are not different ($P < 0.05$); $^{a–d}$means in the same column followed by the same lowercase superscript letters are not different ($P < 0.05$)

Thermal properties

Thermal properties of amylose and amylopectin are shown in Table 2. All kinds of amylose showed melting transitions in the higher temperature range (about 120.0 and 130.0°C). These results indicated that disordered regions were fairly thermostable. However, the melting transition temperature of potato amylose was lower than the reported results of Sievert and Wursch (1993) where the dissociation peak temperature was between 140°C and 155°C. The differences in onset and peak temperatures may have

Table 2. Thermal properties of amylose and amylopectin from different starches

Samples	Temperature (°C)		Thermal enthalpy (J/g)
	onset	peak	
Amylose			
Rice	120.60 ± 1.30^{c}	120.95 ± 0.90^{c}	2.11 ± 0.40^{d}
Corn	129.37 ± 1.52^{a}	129.66 ± 1.10^{a}	3.87 ± 0.32^{c}
Pea	125.66 ± 1.10^{b}	125.98 ± 1.42^{b}	4.62 ± 0.53^{b}
Potato	122.50 ± 1.60^{bc}	123.05 ± 0.92^{bc}	6.33 ± 0.80^{a}
Amylopectin			
Rice	53.13 ± 1.10^{a}	79.04 ± 0.95^{b}	0.89 ± 0.20^{c}
Corn	52.51 ± 1.22^{a}	85.21 ± 1.12^{a}	1.33 ± 0.21^{b}
Pea	52.63 ± 1.41^{a}	80.32 ± 1.30^{c}	1.82 ± 0.20^{d}
Potato	53.88 ± 1.02^{a}	81.24 ± 1.50^{b}	2.81 ± 0.66^{a}

$^{a–c}$means in the same column followed by the same lowercase superscript letters are not different ($P < 0.05$)

Table 3. Onset temperature (°C) of retrograded amylose and amylopectin gel stored for 14 days at 4℃

Storage time	Rice	Corn	Pea	Potato
Amylose				
1	$122.40 \pm 0.45^{a,D}$	$123.61 \pm 0.52^{a,C}$	$124.33 \pm 0.44^{a,B}$	$125.81 \pm 0.60^{a,A}$
2	$121.09 \pm 0.62^{b,C}$	$122.42 \pm 0.34^{b,B}$	$122.11 \pm 0.43^{b,B}$	$123.44 \pm 0.62^{b,A}$
3	$120.02 \pm 0.53^{b,B}$	$122.04 \pm 0.38^{b,A}$	$122.18 \pm 0.56^{bc,A}$	$122.85 \pm 0.59^{b,A}$
5	$120.49 \pm 0.47^{b,B}$	$122.09 \pm 0.50^{b,A}$	$122.72 \pm 0.51^{bc,A}$	$122.12 \pm 0.55^{b,A}$
7	$120.52 \pm 0.42^{b,B}$	$122.12 \pm 0.52^{b,A}$	$122.11 \pm 0.64^{bc,A}$	$122.27 \pm 0.48^{b,A}$
9	$119.05 \pm 0.82^{bc,B}$	$122.58 \pm 0.40^{b,A}$	$122.82 \pm 0.71^{bc,A}$	$122.31 \pm 0.67^{b,A}$
11	$118.85 \pm 0.80^{c,B}$	$121.06 \pm 0.61^{b,B}$	$121.02 \pm 0.62^{bc,B}$	$122.23 \pm 0.58^{b,A}$
14	$118.82 \pm 0.82^{c,B}$	$121.51 \pm 0.32^{b,B}$	$120.01 \pm 0.72^{c,B}$	$122.35 \pm 0.63^{b,A}$
Amylopectin				
1	$40.04 \pm 0.45^{a,D}$	$39.58 \pm 0.52^{a,C}$	$40.14 \pm 0.44^{a,B}$	$41.68 \pm 0.60^{a,A}$
2	$42.86 \pm 0.18^{bc,C}$	$40.02 \pm 0.20^{b,B}$	$43.39 \pm 0.10^{b,B}$	$42.73 \pm 0.20^{b,A}$
3	$43.94 \pm 0.80^{c,B}$	$43.42 \pm 0.68^{b,A}$	$43.55 \pm 0.12^{bc,A}$	$42.68 \pm 0.59^{b,A}$
5	$44.25 \pm 0.27^{c,B}$	$44.53 \pm 0.50^{b,A}$	$44.89 \pm 0.51^{bc,A}$	$43.72 \pm 0.26^{b,A}$
7	$46.10 \pm 0.42^{c,B}$	$43.92 \pm 0.52^{b,A}$	$45.37 \pm 0.85^{bc,A}$	$45.61 \pm 0.48^{b,A}$
9	$48.84 \pm 0.85^{c,B}$	$44.60 \pm 0.40^{b,A}$	$47.10 \pm 0.11^{bc,A}$	$47.10 \pm 0.22^{b,A}$
11	$48.75 \pm 0.26^{c,B}$	$45.62 \pm 0.25^{b,B}$	$47.06 \pm 0.28^{bc,B}$	$47.58 \pm 0.58^{b,A}$
14	$49.67 \pm 0.25^{c,B}$	$46.23 \pm 0.32^{b,B}$	$49.15 \pm 0.98^{c,B}$	$49.33 \pm 0.71^{b,A}$

$^{a–d}$means in the same column followed by the same lowercase superscript letters are not different ($P < 0.05$); $^{A–D}$numbers followed by the same uppercase superscript letters in the same row are not different ($P < 0.05$)

been caused by differences in the concentration of amylose. The melting behaviour of amylose depends on its bound water content (Liu *et al.* 1999). As shown in Tab1e 2, the thermal enthalpy of amylose increased sharply with intrinsic viscosity, and the thermal enthalpy (ΔH_g) was positively correlated to the intrinsic viscosity value ($r = 0.969$, $P < 0.01$) (Table 6). However, the onset and peak temperatures were not related to the intrinsic viscosity of amylose. These results indicate that the granule and crystal characteristics of amylose influence the onset and peak temperature of amylose during heating.

All kinds of amylopectin showed melting transition temperatures in the range of 40°C and 100°C, which indicate that disordered regions were not thermostable. The thermal enthalpy (ΔH_g) of amylopectin increased with intrinsic viscosity (Table 2), and was positively correlated with it ($r = 0.807$, $P < 0.05$) (Table 6). However, the onset and peak temperatures were not related to the intrinsic viscosity, the granule and crystal characteristics of amylopectin may influence the onset and peak temperatures. Moreover, the thermal behaviour of amylose and amylopectin depends mainly on the molecular characteristics and the bound water content (Liu *et al.* 1999).

Retrogradation properties

Retrogradation properties of amylose. Retrogradation properties of amylose stored at 4°C for 14 days were determined by DSC. As shown in Table 3, the onset and peak temperature values of retrograded amylose decreased on the first day, and changed slowly with storage time. These results indicate that amylose retrograded within one day, and later the storage time had no significant effect on the onset and peak temperatures. Moreover, the onset and peak temperature values were positively related to the intrinsic viscosity of amylose during one-day storage. These results indicate that the intrinsic viscosity of amylose is related to the peak temperature value of retrograded amylose. However, no obvious relation to the peak temperature was observed when stored for a long time. As shown in Table 5, the retrogradation enthalpy of amylose increased within two days, and then it increased slowly with storage time. Moreover, the retrogradation enthalpy values of amylose were positively related to its intrinsic viscosity during one-day storage (Table 6), however, no obvious relations to the retrogradation were observed when stored for a long time. From these results, we conclude that the amylose rapidly retrograded in the first 24 h and storage for a long time had small effects on its retrogradation. Similar results were observed by Lu *et al.* (1997) and Ding *et al.* (2003) for potato and rice amyloses, respectively. Furthermore, the retrogradation enthalpy of amylose in the first 24 h was positively related to intrinsic viscosity, which may be due to the molecular weight, as the intrinsic viscosity was positively related to molecular weight (Cowie *et al.*

Table 4. Peak temperature (°C) of retrograded amylose and amylopectin gel stored for 14 days at 4°C

Storage time	Rice	Corn	Pea	Potato
Amylose				
1	122.78 ± 0.48^{a,D}	123.90 ± 0.35^{a,D}	124.54 ± 0.41^{a,B}	126.12 ± 0.62^{a,A}
2	121.56 ± 0.52^{bc,C}	123.33 ± 0.41^{b,C}	122.77 ± 0.54^{b,B}	123.96 ± 0.51^{b,A}
3	120.46 ± 0.53^{c,B}	122.11 ± 0.68^{c,B}	122.83 ± 0.55^{bc,A}	123.21 ± 0.49^{b,A}
5	120.76 ± 0.37^{c,B}	122.94 ± 0.46^{c,B}	123.57 ± 0.41^{bc,A}	122.81 ± 0.53^{b,A}
7	120.87 ± 0.47^{c,B}	122.69 ± 0.52^{c,B}	123.50 ± 0.50^{bc,A}	122.39 ± 0.48^{b,A}
9	119.70 ± 0.85^{c,B}	122.88 ± 0.65^{c,B}	123.14 ± 0.61^{bc,A}	122.66 ± 0.47^{b,A}
11	119.24 ± 0.50^{c,B}	121.30 ± 0.60^{c,B}	121.31 ± 0.60^{bc,B}	122.77 ± 0.78^{b,A}
14	119.10 ± 0.63^{c,B}	122.13 ± 0.72^{c,B}	120.67 ± 0.82^{c,B}	122.95 ± 0.65^{b,A}
Amylopectin				
1	47.41 ± 0.52^{a,C}	53.04 ± 0.52^{a,C}	56.77 ± 0.44^{a,B}	55.73 ± 0.60^{a,A}
2	56.29 ± 0.20^{b,B}	53.03 ± 0.34^{b,B}	57.38 ± 0.38^{b,B}	56.02 ± 0.74^{b,A}
3	56.04 ± 0.75^{c,B}	55.95 ± 0.68^{b,A}	56.30 ± 0.35^{bc,A}	55.05 ± 0.59^{b,A}
5	56.59 ± 0.31^{c,B}	56.15 ± 0.50^{b,A}	56.89 ± 0.51^{bc,A}	55.24 ± 0.23^{b,A}
7	57.54 ± 0.42^{c,B}	55.52 ± 0.52^{b,A}	57.24 ± 0.14^{bc,A}	57.66 ± 0.48^{b,A}
9	59.51 ± 0.85^{c,B}	56.45 ± 0.40^{b,A}	58.10 ± 0.30^{bc,A}	57.65 ± 0.20^{b,A}
11	59.48 ± 0.21^{,B}	56.80 ± 0.23^{b,B}	57.94 ± 0.24^{bc,B}	57.72 ± 0.58^{b,A}
14	60.05 ± 0.64^{c,B}	56.66 ± 0.32^{b,B}	58.91 ± 0.80^{c,B}	58.02 ± 0.23^{b,A}

$^{a–d}$means in the same column followed by the same lowercase superscript letters are not different (P < 0.05); $^{A–D}$numbers followed by the same uppercase superscript letters in the same row are not different (P < 0.05)

1960), i.e. retrogradation enthalpy of amylose in the first 24 h is positively related to molecular weight.

Retrogradation properties of amylopectin. Retrogradation properties of amylopectin stored at 4°C for 14 days were determined by DSC. The onset temperatures of retrograded amylopectin increased with storage time (Table 4). As shown in Table 4, no significant change was observed in the peak tem-

Table 5. Retrogradation enthalpy (J/g) of retrograded amylose and amylopectin gel stored for 14 days at 4°C

Storage time	Rice	Corn	Pea	Potato
Amylose				
1	4.99 ± 0.45^{a,D}	4.04 ± 0.52^{a,C}	3.01 ± 0.44^{a,B}	1.80 ± 0.60^{a,A}
2	5.38 ± 0.62^{bc,C}	4.68 ± 0.34^{b,B}	4.88 ± 0.43^{b,B}	4.54 ± 0.62^{b,A}
3	5.05 ± 0.53^{c,B}	4.56 ± 0.68^{b,A}	4.80 ± 0.55^{bc,A}	4.67 ± 0.59^{b,A}
5	4.76 ± 0.47^{c,B}	4.49 ± 0.50^{b,A}	5.15 ± 0.51^{bc,A}	5.15 ± 0.53^{b,A}
7	5.03 ± 0.42^{c,B}	4.60 ± 0.52^{b,A}	4.94 ± 0.64^{bc,A}	5.11 ± 0.48^{b,A}
9	5.35 ± 0.85^{c,B}	4.68 ± 0.40^{b,A}	5.18 ± 0.71^{bc,A}	5.17 ± 0.67^{b,A}
11	5.77 ± 0.70^{c,B}	4.66 ± 0.61^{b,B}	4.59 ± 0.62^{bc,B}	5.12 ± 0.58^{b,A}
14	5.67 ± 0.61^{c,B}	4.33 ± 0.32^{b,B}	4.83 ± 0.82^{c,B}	5.38 ± 0.63^{b,A}
Amylopectin				
1	0.31 ± 0.45^{a,D}	0.15 ± 0.52^{a,C}	0.10 ± 0.14^{a,B}	0.03 ± 0.10^{a,A}
2	0.42 ± 0.22^{bc,C}	0.32 ± 0.34^{b,B}	3.70 ± 0.10^{b,B}	6.65 ± 0.46^{b,A}
3	2.20 ± 0.34^{c,B}	0.76 ± 0.68^{b,A}	4.93 ± 0.23^{bc,A}	9.51 ± 0.25^{b,A}
5	5.03 ± 0.42^{c,B}	1.05 ± 0.50^{b,A}	5.57 ± 0.51^{bc,A}	5.15 ± 0.53^{b,A}
7	8.12 ± 0.85^{c,B}	3.96 ± 0.52^{b,A}	6.28 ± 0.43^{bc,A}	10.64 ± 0.48^{b,A}
9	8.89 ± 0.65^{c,B}	5.58 ± 0.40^{b,A}	8.18 ± 0.88^{bc,A}	11.50 ± 0.20^{b,A}
11	9.24 ± 0.49^{c,B}	7.42 ± 0.26^{b,B}	9.10 ± 0.32^{bc,B}	11.53 ± 0.58^{b,A}
14	9.61 ± 0.50^{c,B}	8.18 ± 0.32^{b,B}	9.63 ± 0.20^{c,B}	11.68 ± 0.30^{b,A}

$^{a–d}$means in the same column followed by the same lowercase superscript letters are not different (P < 0.05); $^{A–D}$numbers followed by the same uppercase superscript letters in the same row are not different (P < 0.05)

Table 6. Pearson correlation coefficients for the relationship between intrinsic viscosity, thermal enthalpy and retrogradation enthalpy of amylose and amylopectin

Intrinsic viscosity	Thermal enthalpy of amylose (ΔH_g)	Amylose retrogradation enthalpy (ΔH_r) (1 day)	Thermal enthalpy of amylopectin (ΔH_g)	Amylopectin retrogradation enthalpy (ΔH_r)
Amylose	0.969**	−0.930**	–	–
Amylopectin	–	–	0.807*	−0.978* (1 day) 0.686 (3 day) 0.010 (7 day)

*correlation is significant at the 0.05 level (2-tailed); **correlation is significant at the 0.01 level (2-tailed)

perature values of retrograded amylopectin from different starches during the storage period. This result indicates that the intrinsic viscosity of amylopectins has a low relation to the peak temperature of retrograded amylopectin. As shown in Table 5, the retrogradation enthalpy of amylopectin increased with storage time within 7 days, and then changed slowly with storage time. Similar results were observed during the rice amylopectin retrogradation process within 14 days storage (Yu *et al.* 2012b). However, the relationship between amylopectin retrogradation enthalpy and intrinsic viscosity changed with storage time, and amylopectin retrogradation was negatively related to intrinsic viscosity on the first day ($r = -0.978$, $P < 0.01$) (Table 6), and then there were no obvious relations. These results indicated that amylopectin retrogradation depended not only on intrinsic viscosity but also on molecular weight and extent of molecular branching. This kind of retrogradation behaviour was observed in earlier studies (Mua & Jackson 1998; Yu *et al.* 2009, 2012b).

CONCLUSIONS

Thermal and retrogradation properties of amylose and amylopectin were related to intrinsic viscosity. The thermal enthalpy (ΔH_g) values of amylose and amylopectin were positively correlated with the intrinsic viscosity value, however, the onset and peak temperatures were not significantly related to the intrinsic viscosity. The amylose and amylopectin retrogradation enthalpy value was negatively related to intrinsic viscosity, while the onset and peak temperature values of retrograded amylose and amylopectin were not related to the intrinsic viscosity during storage. Therefore, the relationship between intrinsic viscosity and thermal and retrogradation properties of amylose and amylopectin may be useful in developing new applications and processing of starchy foods.

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