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Effects of treatment pressure, holding time, and starch content on gelatinization and retrogradation properties of potato starch–water mixtures treated with high hydrostatic pressure

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Abstract

Potato starch–water mixtures (10–70% w/w) were treated with high hydrostatic pressure (HHP) at 600, 800, and 1000 MPa at 40 °C for 18 and 66 h, and enthalpy change for gelatinization (ΔH_{gel}), gelatinization temperature (T_{gel}), and enthalpy change for re-gelatinization of retrograded starch (ΔH_{retro}) were investigated by differential scanning calorimetry. Furthermore, the results were compared with those of samples HHP-treated for 1 h reported in our previous study. The ΔH_{gel} values of 10–50% (w/w) mixtures decreased with increased treatment pressure and with decreased starch content. The 30–60% (w/w) mixtures retrograded, and the ΔH_{retro} values increased with decreased ΔH_{gel} values and with increased starch content. There was little effect of holding time on the values of ΔH_{gel} and ΔH_{retro}. The T_{gel} values of the 10–50% (w/w) mixtures increased with decreased ΔH_{gel} values and with prolonged holding time.

Keywords: Potato starch; Gelatinization; Retrogradation; High hydrostatic pressure; Differential scanning calorimetry

1. Introduction

There has been considerable interest in the use of high hydrostatic pressure (HHP) treatment for physical modification of starch, because HHP-treated starch shows unique gelatinization and retrogradation properties. For example, pressure-gelatinized starch maintains its granular structure (Douzals, Perrier-Cornet, Gervais, & Coquille, 1998; Stolt, Oinonen, & Autio, 2001; Stute, Klingler, Boguslawski, Eshtiaghi, & Knorr, 1996), and shows a lower quantity of released amylose (Douzals et al., 1998; Stolt et al., 2001) and lower initial rate of enzymatic reactivity (Selmi, Marion, Perrier Cornet, Douzals, & Gervais, 2000) than heat-gelatinized one. In addition, it is reported that retrogradation can be observed immediately after HHP treatment (Hibi, Matsumoto, & Hagiwara, 1993; Katopo, Song, & Jane, 2002; Stute et al., 1996). Although retrogradation is understood to be a typical quality loss of starchy foods, it is of interest to use HHP-treated starch as resistant starch in recent years (Bauer, Wiehle, & Knorr, 2005).

Physical modification of starch–water mixtures with HHP treatment has often been investigated in terms of treatment pressure, treatment temperature, and holding time. At a constant temperature and time, degree of gelatinization of starch–water mixtures increases with increase in treatment pressure and decrease in starch content (Katopo et al., 2002; Kawai, Fukami, & Yamamoto, 2007; Muhr & Blanshard, 1982). In our previous study (Kawai et al., 2007), gelatinization and retrogradation properties of 10–70% (w/w) potato starch–water mixtures which were HHP-treated at 40 °C for 1 h were investigated by DSC (differential scanning calorimetry). Gelatinization pressure, treatment

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pressure required for complete gelatinization, increased with increased starch content, and 10–50% (w/w) mixtures were gelatinized completely by HHP treatment in the pressure range from 700 to 1200 MPa. In addition, it was found that 30–60% (w/w) mixtures showed retrogradation. These observations were summarized as a state diagram of treatment pressure vs. starch content.

It has been known that degree of gelatinization of HHP-treated starch depends on treatment temperature. Douzals, Perrier-Cornet, Coquille, and Gervais (2001) demonstrated that the degree of gelatinization of 5% (w/w) wheat starch suspension increased with increased treatment temperature in the range between 25 °C and its heat gelatinization temperature, and with decreased temperature in the range between −20 and 25 °C. Similarly, Bauer and Knorr (2005) reported that degrees of gelatinization of 5% (w/w) wheat, potato, and tapioca starch suspensions increased with increased treatment temperature in the range between 25 °C and their heat gelatinization temperatures. On the other hand, Rubens and Heremans (2000) demonstrated that there was little effect of treatment temperature on gelatinization pressure of 10 mg/ml (approximately 1% w/w) rice starch suspension in the range between −5 and 45 °C. These results were summarized as state and/or phase diagrams of pressure vs. temperature (Bauer & Knorr, 2005; Douzals et al., 2001; Rubens & Heremans, 2000).

There have been many studies on pressure gelatinization of starch–water mixtures which were HHP-treated for up to 0.25 h (Błaszczak, Fornal, Valverde, & Garrido, 2005a; Błaszczak, Valverde, & Fornal, 2005b; Douzals et al., 1998, 2001; Selmi et al., 2000; Stute et al., 1996). Stolt et al. (2001) and Bauer and Knorr (2004, 2005) demonstrated that degree of gelatinization increased significantly with prolonged holding time in the range up to 1 h. Even at longer holding times than 1 h, there was relatively significant effect of holding time on pressure gelatinization of 5% (w/w) wheat starch suspension treated at 350 MPa at 29 °C. On the other hand, 5% (w/w) potato starch suspension treated at 700 MPa at 29 °C showed little change in the degree of gelatinization upon HHP treatment in the range between 1 and 4 h.

It has previously been shown that various experimental conditions (pressure, temperature, and time) and composition of starch–water mixture (botanical sources of starch and starch content) affected gelatinization and retrogradation properties of HHP-treated starch. So far, studies on the effects of treatment pressure and temperature have been conducted extensively. However, data on the effect of the holing time is not limited. Actually, HHP treatment for tens of hours, which is expressed as long-time HHP treatment in this paper, has never been employed for the physical modification of starch. This could be because long-time HHP treatment is judged to be too extensive to be practical on an industrial scale. A study of long-time HHP treatment, is however, of importance in understanding the effect of HHP treatment on starch. Potato starch is more resistant to pressure than other starches (Bauer, Hartmann, Sommer, & Knorr, 2004; Błaszczak et al., 2005a, 2005b; Douzals, Marechal, Coquille, & Gervais, 1996; Hibi et al., 1993; Katopo et al., 2002; Muhr & Blanshard, 1982; Selmi et al., 2000; Stute et al., 1996), and long-time HHP treatment may affect pressure gelatinization properties of potato starch. In addition, it was suggested that retrogradation could occur "during" HHP treatment (Katopo et al., 2002; Stute et al., 1996). If so, it is expected that degree of retrogradation would increase with prolonged holding time.

The purpose of this study was to investigate the effects of treatment pressure, holding time for tens of hours, and starch content on gelatinization and retrogradation properties of potato starch–water mixtures treated with HHP. In the current study, treatment pressures of 600, 800, and 1000 MPa, treatment temperature of 40 °C, and holding times of 18 and 66 h were employed, and thermal properties of the HHP treated samples (10–70% w/w) were evaluated by DSC. In order to compare the results with those of the samples which were HHP-treated at 40 °C for 1 h reported in our previous study (Kawai et al., 2007), the treatment temperature at 40 °C was employed in this study.

2. Materials and methods

2.1. Preparation of sample

Potato starch was purchased from Hokuren, Japan. The initial moisture content was gravimetrically determined to be 19.7% (w/w) by oven-drying at 105 °C for 12 h. The starch (50–200 mg) and distilled water were put into a polymer pouch (7 × 2 cm), and heat-sealed to prepare starch–water mixtures (50%, 60%, and 70% w/w). For preparing 10–40% (w/w) mixtures, 2% (w/w) heat-gelatinized waxy corn starch was used instead of water in order to prevent the sedimentation of potato starch during pressure treatment (Kawai et al., 2007; Stolt et al., 2001). In our preliminary experiment, it was confirmed visually that phase separation between gelatinized starch and bulk water was induced by the sedimentation of potato starch during HHP treatment and that it was prevented by using the heat-gelatinized waxy corn starch as a thickener. The pouches containing 60% and 70% (w/w) mixtures were preserved at ambient temperature for a few days in order to minimize its inhomogeneity of water before setting in HHP-generating system. It was confirmed in preliminary experiments that the preservation procedure did not affect the thermal properties of the samples. The pouches containing 10–50% (w/w) mixtures were shaken manually and put in the HHP-generating apparatus.

2.2. High hydrostatic pressure treatment

The sample was treated with a HHP-generating system (HPS-1400: Teramecs Co. Ltd., Japan). A castor oil and ethanol mixture (volume ratio of 1 : 1) was used as a pressure medium. The temperature of pressure medium was kept at 40 °C with a heat controller. The temperature changes during the build-up and release of pressure were not measurable because of the limitations of experimental setting. Pouched sample was put into the HHP-vessel (2 cm O × 8 cm) containing the pressure medium. Pressure was built up at approximately 100 MPa/min and held at the treatment pressure of 600, 800, and 1000 MPa for 18 and 66 h. The treatment pressure was kept within the variation of ±5% by an automatic pressure control system built in the machine. The pressure was released at approximately 100 MPa/min, and then thermal properties of the HHP-treated samples were investigated by DSC.

2.3. *Differential scanning calorimetry*

Thermal properties (ΔH_{gel}; enthalpy change for heat gelatinization, T_{gel}; gelatinization temperature, and ΔH_{retro}; enthalpy change for re-gelatinization of retrograded starch) of HHP-treated starch were investigated by a DSC (Diamond DSC: Perkin-Elmer Co. Ltd., USA). An empty stainless steal pan was used as a reference, and the temperature and heat flow were calibrated with indium and distilled water. Sample (10–30 mg) was put into the steal pan, and then the starch content was adjusted below 30% (w/w) by adding distilled water. DSC measurement were performed at 10 °C/min in the temperature range from 10 to 120 °C. The DSC thermogram was analyzed by using Thermal Analysis Pyris Software interfaced with the DSC. The values of ΔH_{gel} and T_{gel} were evaluated from the peak area and the onset point in the endothermic peak of gelatinization at about 60 °C, respectively. The value of ΔH_{retro} was evaluated from the peak area in the broad endothermic peak corresponding to re-gelatinization of retrograded starch at about 40 °C. In the case that the endothermic peaks of the heat gelatinization and re-gelatinization of retrograded starch overlapped, both ΔH_{gel} and ΔH_{retro} were evaluated by deconvolution of the overlapped curve into two endothermic peaks with the analytical software interfaced with the DSC (Kawai et al., 2007).

3. Results and discussion

3.1. *DSC thermogram for potato starch–water mixtures treated with HHP*

Four types of DSC thermograms were observed, and the typical thermograms including a typical reference thermogram were shown in Fig. 1: DSC thermograms for 10%, 40%, 50%, and 60% (w/w) starch–water mixtures treated at 1000 MPa for 66 h and 30% (w/w) native potato starch–water mixture. The non-treated 30% (w/w) mixture showed an endothermic peak of gelatinization at about 60 °C, and then ΔH_{gel} and T_{gel} were evaluated from the area and the onset temperature of the peak, respectively. As similar to the non-treated 30% (w/w) mixture, the HHP-treated 60% (w/w) mixture showed an endothermic peak of gelatinization, and then ΔH_{gel} and T_{gel} were evaluated. In contrast, the HHP-treated 10% (w/w) mixture did not show any peak because of its complete pressure gelatinization. On the other hand, the HHP-treated 40% (w/w) mixture showed a broad endothermic peak as a consequence of re-gelatinization of retrograded starch at about 40 °C, and then ΔH_{retro} was evaluated from the area of the broad peak. The HHP-treated 50% (w/w) mixture showed an overlapped curve due to re-gelatinization of retrograded starch and gelatinization. In this case, ΔH_{gel}, T_{gel}, and ΔH_{retro} were evaluated by deconvolution of the curve into two endothermic peaks. In the following sections, the values of ΔH_{gel}, T_{gel}, and ΔH_{retro} were compared with those of the samples HHP-treated for 1 h (Kawai et al., 2007), and the effects of treatment pressure, holding time for tens of hours, and starch content on the values of ΔH_{gel}, T_{gel}, and ΔH_{retro} were discussed.

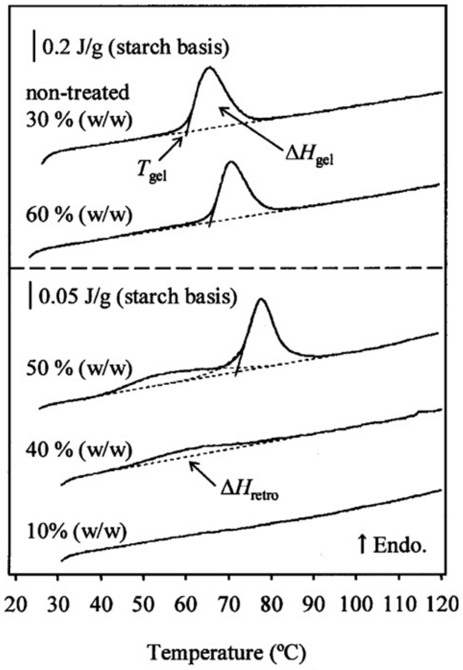

Fig. 1. DSC thermograms for 30%, 40%, 50%, and 60% (w/w) potato starch–water mixtures HHP-treated at 1000 MPa for 66 h and 30% (w/w) native potato starch–water mixture.

3.2. *Gelatinization of HHP-treated potato starch–water mixtures*

Fig. 2 shows the effect of HHP treatment on ΔH_{gel} of 10–70% (w/w) starch–water mixtures. The ΔH_{gel} values decreased with decreased starch content and increased treatment pressure, and 10–40% (w/w) mixtures were gelatinized completely by HHP treatment at up to 1000 MPa. The results agreed roughly with those in previous reports (Bauer & Knorr, 2005; Katopo et al., 2002; Muhr & Blanshard, 1982). However, it should be noted that 2% (w/w) heat-gelatinized waxy corn starch which was used instead of water as a thickener might have reduced the impact of HHP treatment on gelatinization because of its elevated viscosity as pointed out by Bauer and Knorr (2005). In addition, temperature increase during the build-up of pressure, which would depend on HHP-generating systems,

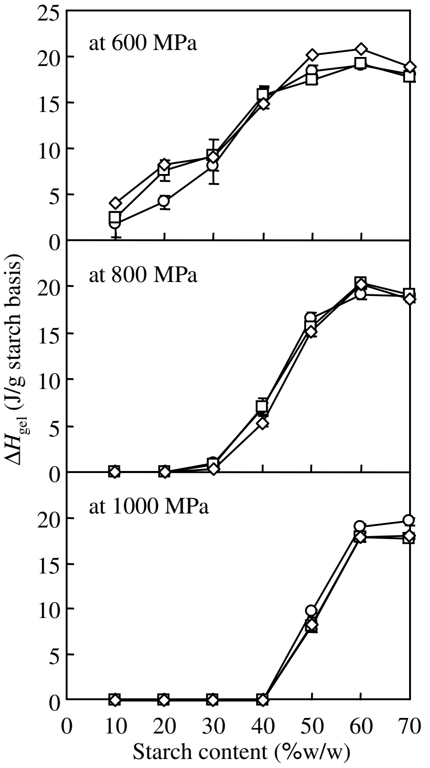

Fig. 2. ΔH_{gel} versus starch content for 10–70% (w/w) potato starch–water mixtures HHP-treated at 600, 800, and 1000 MPa for 1 h (circle), 18 h (square), and 66 h (diamond).

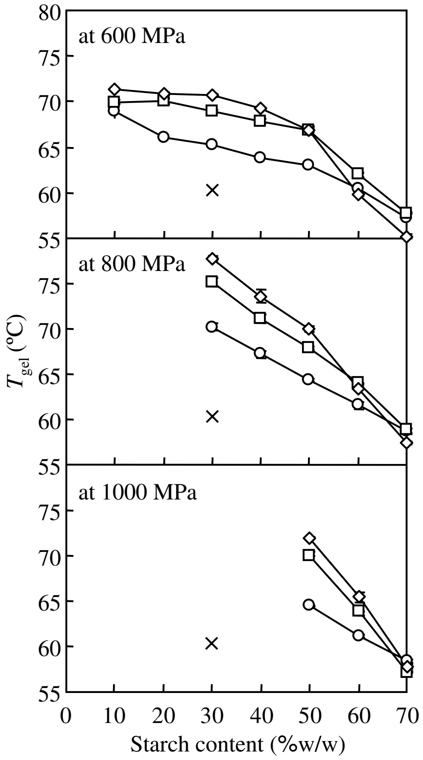

Fig. 3. T_{gel} versus starch content for 10–70% (w/w) potato starch–water mixtures HHP-treated at 600, 800, and 1000 MPa for 1 h (circle), 18 h (square), and 66 h (diamond). The cross symbol indicates the result for 30% (w/w) native potato starch–water mixture.

might have temporarily promoted progress of pressure gelatinization because it was reported that a higher holding temperature promoted pressure gelatinization of potato starch water mixtures (Bauer & Knorr, 2005). Furthermore, it was pointed out that the methods to evaluate gelatinization (microscopic study and DSC study) affect the results (Bauer & Knorr, 2005; Douzals et al., 2001). Therefore, the properties of pressure gelatinization may not be completely comparable between their reports and ours.

It was found that the ΔH_{gel} values were independent of holding time in the time range between 1 and 66 h. As has been reviewed above, it is reported that the effect of holding time longer than 1 h on pressure gelatinization are affected by the treatment conditions (pressure and temperature) and the composition of starch–water mixture (botanical sources of starch and starch content). For example, in the time range between 1 and 4 h, 5% (w/w) wheat starch suspension treated at 350 MPa at 29 °C and 5% (w/w) tapioca starch suspension treated at 530 MPa at 29 °C showed, respectively, a major increase and a small increase in the degree of gelatinization (Bauer & Knorr, 2005). In addition, 25% (w/w) barley starch suspension treated at 400 MPa at 30 °C showed a little decrease in ΔH_{gel} value in the time range between 1 and 1.25 h. In the case of potato starch–water mixture, it was reported that 5% (w/w) potato starch suspension treated at 700 MPa at 29 °C showed little increase in the degree of gelatinization in the range between 1 and 4 h (Bauer & Knorr, 2005). In this study, it was demonstrated that HHP treatment in the time range between 1 and 66 h showed little effect on ΔH_{gel} which was independent of treatment pressure (600, 800, and 1000 MPa) and starch content (10–70% w/w). It can be concluded that pressure gelatinization of potato starch–water mixtures will not be promoted by prolonging holding time of HHP treatment.

3.3. Gelatinization temperature of HHP-treated potato starch–water mixtures

Fig. 3 shows the effect of HHP treatment on T_{gel} of 10–70% (w/w) starch–water mixtures. For comparison, the result of a non-treated 30% (w/w) mixture was plotted. At each treatment pressure, the T_{gel} value increased with decreased starch content. In addition, 10–50% (w/w) mixtures showed increase in T_{gel} with prolonged holding time, although the effect of holding time on T_{gel} of 60% (w/w) mixture was not significant. The T_{gel} value of 70% (w/w) mixture was lower than that of non-treated one. In comparison with the results in Figs. 2 and 3, it should be noted that the increase in T_{gel} will be related to the progress of pressure gelatinization (decrease in ΔH_{gel}).

Based on this relationship, T_{gel} values of HHP-treated mixtures were plotted against ΔH_{gel} values at each treatment pressure in Fig. 4. It was confirmed that T_{gel} values increased with decreased ΔH_{gel} values in the starch content range between 10 and 60% (w/w). In addition, the T_{gel} values of 10–50% (w/w) mixtures increased with prolonged

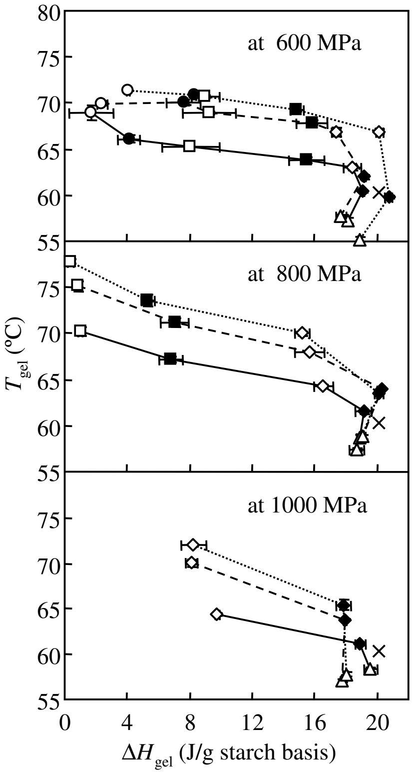

Fig. 4. T_{gel} versus ΔH_{gel} for 10–70% (w/w) potato starch–water mixtures HHP-treated at 600, 800, and 1000 MPa for 1 h (solid line), 18 h (broken line), and 66 h (dotted line). The symbol indicates the starch content of the mixtures: open circle, 10%; closed circle, 20%; open square, 30%; closed square, 40%; open diamond, 50%; closed diamond, 60%; open triangle, 70%. The cross symbol indicates the result for 30% (w/w) native potato starch–water mixture.

holding time. The treatment pressure did not affect the dependence of ΔH_{gel} values on T_{gel} significantly.

It has been widely recognized that heat gelatinization of starch in the presence of excess water is a cooperative melting of crystalline and amorphous regions in starch granules; destabilization of amorphous region is induced by water absorption and it triggers the melting of crystalline regions (Jacobs et al., 1998; Tester & Debon, 2000). In addition, the amorphous region in starch granules increases with increase in the degree of gelatinization. Taking into consideration these interpretations, it is suggested that the increased amorphous region suppresses the melting of remained crystalline regions in the starch granules because water absorption and swelling are delayed due to restricted water diffusion in the amorphous region. Consequently, T_{gel} values increased with the decrease in the ΔH_{gel} values (Fig. 4). The reason why the T_{gel} values of HHP-treated 70% (w/w) mixture were lower than that of non-treated one was unclear at this stage. However, similar results are also reported by Katopo et al. (2002); HHP-treated low moisture starches (maize, high amylose maize, waxy maize, tapioca, rice, potato containing 84–90% (w/w) starch) showed lower T_{gel} values than non-treated ones.

Annealing causes a shift of T_{gel} to higher temperatures (Jacobs et al., 1998; Tester & Debon, 2000; Waduge, Hoover, Vasanthan, Gao, & Li, 2006). Annealing is an isothermal holding treatment of starch containing excess or intermediate amount of water at temperatures below the gelatinization temperature (T_{gel}). When thermal properties of annealed starch were investigated by DSC, shift of T_{gel} to higher temperatures and unchanged or higher ΔH_{gel} were observed (Jacobs et al., 1998; Tester & Debon, 2000; Waduge et al., 2006). In fact, we confirmed that potato starch–water mixtures (10–70% w/w) annealed at ambient pressure at 40 °C for 66 h showed increase in T_{gel} by 4.0 °C and increase in ΔH_{gel} by 2.8 J/g. Although the mechanism of annealing has not been completely understood, it is suggested that the amorphous region becomes more rigid and less mobile within annealed starch granules, and this event delays the destabilization of the amorphous region induced by water absorption and swelling (Tester & Debon, 2000). Taking into account the suggested mechanism of annealing, long-time HHP treatment may make amorphous regions more rigid and less mobile as the holding time is prolonged. This will be supported by the fact that the T_{gel} values of 10–50% (w/w) mixtures increased with prolonged holding time. Treatment pressure did not affect the dependence of ΔH_{gel} values on T_{gel} significantly, therefore, it is suggested that T_{gel} will be elevated at lower treatment pressures than 600 MPa.

3.4. Retrogradation of HHP-treated potato starch–water mixtures

Fig. 5 shows the effect of HHP treatment on ΔH_{retro} of 10–70% (w/w) starch–water mixtures. It was found that HHP-treated 30–50% (w/w) mixtures showed retrogradation depending on the treatment pressure and starch content. In comparison with the results in Figs. 2 and 5, it was noted that retrogradation was observed when the mixtures gelatinized at least partially. This indicates that a certain amount of the gelatinized part of starch granules induced by HHP treatment was required for the progress of retrogradation. In addition, it was confirmed that the tendency of retrogradation observed in HHP-treated mixtures was in good agreement with that of heat-gelatinized starch; heat gelatinized starch retrograded in the starch content range between 30 and 60% (w/w), and degree of retrogradation increased with increased starch content in the starch content range (Miles, Morris, Orford, & Ring, 1985; Orford, Ring, Carroll, Miles, & Morris, 1987).

The effect of holding time on the ΔH_{retro} values was not significant. It has been reported that retrogradation is observed immediately after HHP treatment (Hibi et al., 1993; Katopo et al., 2002; Stute et al., 1996), although the mechanism has been unclear. Stute et al. (1996) suggested that retrogradation might have occurred “during” HHP treatment. If so, ΔH_{retro} value would increase with prolonged holding time. However, there was no apparent correlation between the ΔH_{retro} values and the holding time in this study. This result suggests that retrogradation progressed immediately after HHP treatment rather than

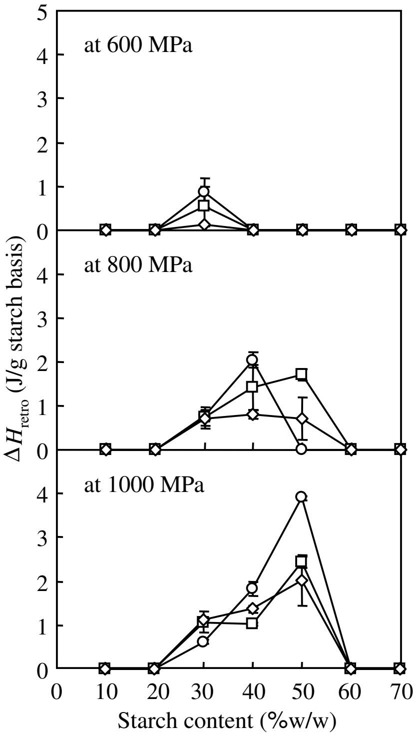

Fig. 5. ΔH_{retro} versus starch content for 10–70% (w/w) potato starch–water mixtures HHP-treated at 600, 800, and 1000 MPa for 1 h (circle), 18 h (square), and 66 h (diamond).

during the treatment. Taking the time of our experimental procedure into consideration, it was estimated that the ΔH_{retro} value corresponded to the progress of retrogradation at least within a few hours of HHP treatment. In addition, it should be noted that temperature decreases during release of pressure, because retrogradation is promoted generally at low temperatures above its freezing temperature (Colwell, Axford, Chamberlain, & Elton, 1969; Hoover, 1995). In order to understand the mechanism of retrogradation of HHP-treated starch in more detail, a further study on the relationship between processing temperature and time within and after HHP treatment and degree of retrogradation (*in situ* real-time measurement of retrogradation within and after HHP treatment) will be necessary.

4. Conclusion

Although it has been reported that various experimental conditions (pressure, temperature, and time) and composition of starch–water mixture (botanical sources of starch and starch content) affected gelatinization and retrogradation properties of starch–water mixtures treated with HHP, published data was not sufficient to understand it completely. In this study, effects of treatment pressure (600, 800, and 1000 MPa), holding time (18 and 66 h), and starch content (10–70% w/w) on gelatinization and retrogradation properties of HHP-treated potato starch–water mixtures were investigated. It was demonstrated that the values of ΔH_{gel} and ΔH_{retro} were affected significantly by treatment pressure and starch content, though there was little effect of holding time. In addition, it was found that the T_{gel} values increased with decreased ΔH_{gel} values and with prolonged holding time. Although the long-time HHP treatment is impractical from the viewpoint of running cost in starch industry, it is of fundamental importance to understand gelatinization and retrogradation properties of HHP-treated starch. A possible extension of this research would involve further collection of fundamental data on gelatinization and retrogradation properties, clarification of their mechanisms, investigation of other physico-chemical properties (*e.g.*, swelling index and the maximum degree of hydration), and proposal for practical use of HHP-treated starch.

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