

State diagram of potato starch–water mixtures treated with high hydrostatic pressure

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Abstract

Potato starch–water mixture was treated with high hydrostatic pressure (HHP) of up to 1.2 GPa, and effect of starch content (10–70% (w/w)) on HHP-gelatinization was investigated by differential scanning calorimetry (DSC). Depending on the treatment pressure and potato starch content, DSC thermograms showed decrease in enthalpy change of heat gelatinization reflecting the progress of HHP-gelatinization and increase in enthalpy change of re-gelatinization of retrograded starch. From the viewpoint of the enthalpy changes, physically modified state of HHP-treated potato starch–water mixtures was classified as follows: no change, partial gelatinization, complete gelatinization, partial gelatinization and retrogradation, and complete gelatinization and retrogradation. A state diagram of potato starch–water mixtures (treatment pressure vs. starch content) was presented.

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1. Introduction

Gelatinization of starch has been achieved traditionally by heating starch with water. Heat-gelatinized starch has various characteristic properties as a food substance and biomass: improvements of digestibility and enzymatic reactivity, and increases in dynamic viscosity and water holding capacity. Heat-gelatinized starch, however, loses gradually the properties depending on the storage time and temperature. This event is recognized as “retrogradation”. While retrogradation has been often treated as a typical quality loss of starchy foods, it is of interest for the use as resistant starch in recent years (Bauer, Wiehle, & Knorr, 2005; Chung, Lim, & Lim, 2006; Escarpa, González, Mañas, García-Diz, & Saura-Calixto, 1996). In order to produce various functional starches, many efforts have been devoted.

The gelatinization of starch can be also achieved by high hydrostatic pressure (HHP) treatment. The use of HHP-

treatment for physical modifications of starch has attracted much attention mainly because HHP-gelatinized starch has different gelatinization and retrogradation properties from heat-gelatinized one. For example, HHP-gelatinized starch maintains its granular structure (Douzals, Perrier-Cornet, Gervais, & Coquille, 1998; Stolt, Oinonen, & Autio, 2001; Stute, Klingler, Boguslawski, Eshtiaghi, & Knorr, 1996), and has lower quantity of released amylose than heat-gelatinized one (Douzals et al., 1998; Stolt et al., 2001). HHP-gelatinized starch also shows a lower initial rate of enzymatic reactivity (Selmi, Marion, Perrier Cornet, Douzals, & Gervais, 2000) and swelling ability (Douzals et al., 1998) than heat-gelatinized one. Additionally, there are some studies that retrogradation was observed immediately after HHP-treatment (Hibi, Matsumoto, & Hagiwara, 1993; Katopo, Song, & Jane, 2002; Stute et al., 1996). The immediate retrogradation will be related to decrease in enzymatic digestibility of HHP-gelatinized starch reported by an earlier study (Hayashi & Hayashida, 1989).

It has been known that the progress of HHP-gelatinization of starch–water mixtures depends markedly on the

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botanical sources of starch, starch content, treatment pressure, temperature, and time. The degree of gelatinization increases with increased treatment pressure; most starches (e.g., corn, waxy corn, rice, wheat, and pie) gelatinize completely by HHP-treatment of up to approximately 0.6 GPa (Bauer, Hartmann, Sommer, & Knorr, 2004; Bauer & Knorr, 2005; Błaszczak, Fornal, Valverde, & Garrido, 2005; Douzals, Marechal, Coquille, & Gervais, 1996; Douzals, Perrier-Cornet, Coquille, & Gervais, 2001; Douzals et al., 1998; Muhr & Blanshard, 1982; Rubens & Heremans, 2000; Stolt et al., 2001; Stute et al., 1996). Potato starch, however, is more resistant to pressure than other starches (Bauer et al., 2004; Błaszczak et al., 2005; Błaszczak, Valverde, & Fornal, 2005; Douzals et al., 1996; Hibi et al., 1993; Katopo et al., 2002; Muhr & Blanshard, 1982; Muhr, Wetton, & Blanshard, 1982; Selmi et al., 2000; Stute et al., 1996). It is reported that 5% (w/w) potato starch suspension below 50 °C did not show complete gelatinization even at 0.7 GPa (Bauer & Knorr, 2005). Gelatinization pressure, treatment pressure required for complete gelatinization, is suggested to be 0.8 GPa for 12.8% potato starch suspension (Muhr & Blanshard, 1982). Treatment temperature and time also affect the HHP-gelatinization significantly. The degree of gelatinization increases with increased treatment temperature in the temperature range between 25 °C and heat gelatinization temperature (Bauer & Knorr, 2005; Douzals et al., 2001; Muhr & Blanshard, 1982). The degree of gelatinization increases significantly with increased treatment time in the range up to 60 min. HHP-treatment for longer than 60 min shows a small effect on the degree of gelatinization (Bauer & Knorr, 2005; Bauer et al., 2004; Stolt et al., 2001). Furthermore, effect of starch content on HHP-gelatinization of starch–water systems is remarkable; the degree of gelatinization increases with decreased the starch content (Katopo et al., 2002; Muhr & Blanshard, 1982).

As above-mentioned, the effects of treatment pressure, temperature, and time on HHP-gelatinizations have been extensively investigated, and the degree of gelatinization has been summarized as the time courses (Bauer & Knorr, 2004, 2005; Stolt et al., 2001) and/or as the pressure–temperature diagrams (Bauer & Knorr, 2005; Douzals et al., 2001; Rubens & Heremans, 2000). In contrast, there has been little study on the effect of starch content on HHP-gelatinization of starch–water mixtures, and thus, there was no systematic experimental conclusion such as a state diagram (treatment pressure vs. starch content). Additionally, little is known on the properties of HHP-gelatinization of potato starch. Most of the studies were carried out with treatment pressures of up to 0.6 GPa (Bauer & Knorr, 2004; Bauer et al., 2004; Błaszczak et al., 2005; Douzals et al., 1996, 1998, 2001; Hayashi & Hayashida, 1989; Hibi et al., 1993; Muhr et al., 1982; Rubens & Heremans, 2000; Selmi et al., 2000; Stolt et al., 2001; Stute et al., 1996) or up to 0.8 GPa (Bauer & Knorr, 2005; Błaszczak et al., 2005; Katopo et al., 2002), thus little has been investigated on HHP-gelatinization of potato starch–water mixtures. Although a high-pressure system generating pressure of up

to 1.5 GPa (Muhr & Blanshard, 1982) had been employed to investigate the HHP-gelatinization of potato starch–water mixtures, experimental conditions such as starch content and treatment pressure were rather limited.

The purpose of this study was to understand the effect of starch content of potato starch–water mixtures on the HHP-gelatinization. HHP-gelatinization of 10–70% (w/w) potato starch–water mixtures treated at treatment pressure of up to 1.2 GPa at a constant temperature for a constant treatment time was investigated with a differential scanning calorimetry (DSC), and then a state diagram (treatment pressure vs. starch content) of potato starch–water mixtures was presented.

2. Materials and methods

2.1. Sample preparation

Commercial potato starch was purchased from Hoku-ren, Japan. The moisture content of the starch was gravimetrically determined to be 19.7% (w/w) by oven-drying at 105 °C for 12 h. The starch (50–200 mg) was put into a polymer pouch (7 × 2 cm), and then distilled water was added to prepare 50–70% starch–water mixtures. The pouch containing the starch–water mixture was heat-sealed and preserved at ambient temperature for a few days in order to minimize inhomogeneity of the mixtures. For preparing 10–40% potato starch mixtures, 2% (w/w) heat-gelatinized waxy corn starch–water mixture was used instead of water in order to prevent the sedimentation of potato starch during pressure treatment (Stolt et al., 2001). The starch suspension sealed in a pouch was shaken manually and set quickly in a HHP-generating system.

2.2. HHP-treatment

HHP-treatment was performed in the pressure range of 0.4–1.2 GPa with a HHP-generating system (HPS-1400: Teramecs Co. Ltd., Japan). A castor oil–ethanol mixture (approximately 50% (v/v)) was used as a pressure medium. The temperature of pressure medium was kept at 40 °C with an equipped temperature controller in order to avoid the crystallization of water in the sample during HHP-treatment, because pure water pressurized at 1.2 GPa turns into ice VI at a temperature below 40 °C (Smeller, 2002). Pouched potato starch–water mixture was put into the HHP-vessel containing the pressure medium, and then HHP-treatment was carried out. Pressure was built up at approximately 0.1 GPa/min and held at a treatment pressure for 60 min. After the HHP-treatment, the pressure was released to ambient pressure at 0.1 GPa/min, and the progress of HHP-gelatinization was investigated by using a DSC.

2.3. DSC measurement

DSC measurement was carried out with a DSC (Diamond DSC: Perkin-Elmer Co. Ltd., USA). Empty stainless

steal pan was used as a reference, and the temperature and heat flow change were calibrated with indium and distilled water. Potato starch–water mixture (approximately 300 mg) was put into the steal pan, and then the starch content was adjusted below 30% (w/w) by adding distilled water. The DSC measurement was performed at 10 °C/min in the temperature range of 10–120 °C. DSC thermogram was analyzed by using Thermal Analysis Pyris Software interfaced with the DSC.

3. Results

DSC thermograms for HHP-treated potato starch–water mixtures showed various characteristic thermal behaviors depending on the treatment pressure and starch content. As typical results, DSC thermograms for 10% mixtures treated at 0.4–0.7 GPa and those for 50% mixtures treated at 0.9–1.2 GPa were shown in Figs. 1 and 2, respectively. The DSC thermogram for the 10% mixture treated at 0.4 GPa showed an endothermic peak ascribed to heat gelatinization, and enthalpy change of gelatinization (ΔH_{gel}) was comparable to that of no-treated sample shown as a reference. Although the mixtures treated at 0.5 and 0.6 GPa also showed heat gelatinization peaks, the values of ΔH_{gel} decreased with increased treatment pressure. The heat gelatinization peak disappeared with HHP-treatment at 0.7 GPa. On the other hand, the DSC thermograms for 50% mixtures showed relevant endothermic peaks due to heat gelatinization as shown in Fig. 2. The peak area indicating ΔH_{gel} decreased with increased treatment pressure. In addition, a shoulder was observed on the peak simultaneously at a lower temperature than the heat gelatinization temperature. By HHP-treatment at 1.2 GPa, the heat gelatinization peak disappeared completely and the shoulder developed into a broad endothermic peak. It should be noted that the thermal event at the low temperature is due to re-gelatinization of retrograded starch (Stute et al., 1996). The DSC

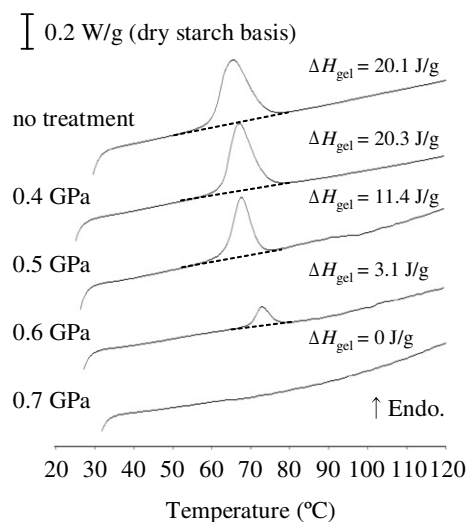


Fig. 1. DSC thermograms for 10% potato starch–water mixtures HHP-treated at 0.4–0.7 GPa and 30% non-treated potato starch–water mixture.

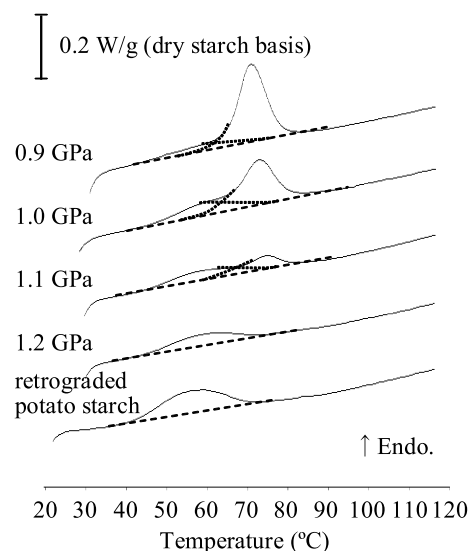


Fig. 2. DSC thermograms for 50% potato starch–water mixtures HHP-treated at 0.9 to 1.2 GPa and a 30% retrograded potato starch–water mixture prepared by storing the heat-gelatinized one for 7 days at 4 °C.

thermogram for retrograded 30% starch–water mixture prepared by keeping the heat-gelatinized one at 4 °C for 7 days was shown there for comparison. Enthalpy change of re-gelatinization of retrograded starch (ΔH_{retro}) was evaluated in order to analyze the progress of retrogradation. 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 as shown in Fig. 2 (dotted line). Although not shown here, ΔH_{gel} and ΔH_{retro} of the other HHP-treated samples were evaluated likewise.

The values of ΔH_{gel} and ΔH_{retro} were plotted against the treatment pressure in Figs. 3 and 4, respectively. From Fig. 3, it was confirmed that ΔH_{gel} values decreased with

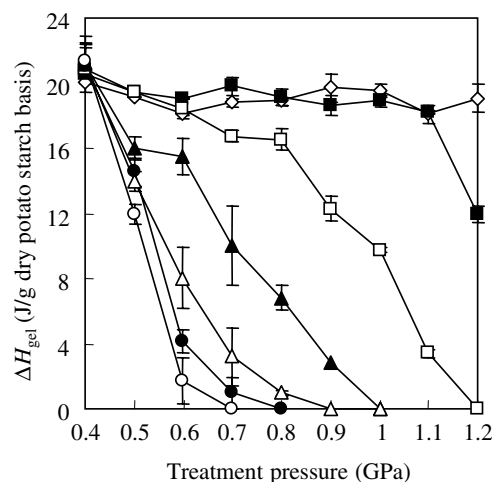


Fig. 3. ΔH_{gel} versus treatment pressure for each potato starch–water mixture: open circle, 10%; closed circle, 20%; open triangle, 30%; closed triangle, 40%; open square, 50%; closed square, 60%; open diamond, 70%.

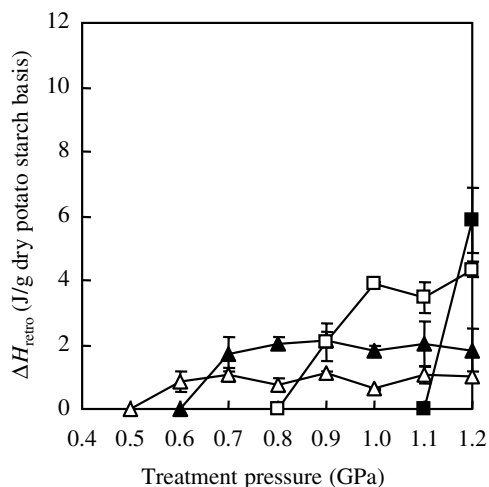


Fig. 4. ΔH_{retro} versus treatment pressure for each potato starch–water mixture: open triangle, 30%; closed triangle, 40%; open square, 50%; closed square, 60%.

increased treatment pressure and decreased starch content. Since no-treated starch showed $\Delta H_{\text{gel}} = 20 \pm 2$ J/g, HHP-treated samples showing $\Delta H_{\text{gel}} < 18$ J/g were judged as partially or completely gelatinized ones. The mixtures of 10–50% showed $\Delta H_{\text{gel}} = 0$ J/g after HHP-treatment at lower than 1.2 GPa. At 1.2 GPa HHP-treated 60% mixture showed a decrease in ΔH_{gel} of about 12 J/g. All the HHP-treated 70% mixtures showed $\Delta H_{\text{gel}} \geq 18$ J/g. On the other hand, from Fig. 4, it was found that 30–60% mixtures retrograded. The higher the starch content was, retrogradation was observed at higher treatment pressure. At each starch content, ΔH_{retro} reached a plateau value and the values increased with increased starch content. Furthermore, in comparison with the results in Figs. 3 and 4, it was found that retrogradation was observed when ΔH_{gel} was below 12 J/g.

From the changes of ΔH_{gel} and ΔH_{retro} , the physically modified states of HHP-treated potato starch–water mixtures were classified as follows: no change ($\Delta H_{\text{gel}} \geq 18$ J/g and $\Delta H_{\text{retro}} = 0$ J/g), partial gelatinization ($\Delta H_{\text{gel}} < 18$ J/g and $\Delta H_{\text{retro}} = 0$ J/g), complete gelatinization ($\Delta H_{\text{gel}} = 0$ J/g and $\Delta H_{\text{retro}} = 0$ J/g), partial gelatinization and retrogradation ($\Delta H_{\text{gel}} < 18$ J/g and $\Delta H_{\text{retro}} > 0$ J/g), and complete gelatinization and retrogradation ($\Delta H_{\text{gel}} = 0$ J/g and $\Delta H_{\text{retro}} > 0$ J/g). The classifications were presented as a state diagram of potato starch water mixture (treatment pressure vs. starch content) in Fig. 5.

4. Discussion

4.1. Gelatinization of HHP-treated potato starch–water mixtures

Although there have been many studies on HHP-gelatinization of potato starch–water mixtures, little has been known on the gelatinization pressure because potato starch is much more resistant to pressure than the starches of

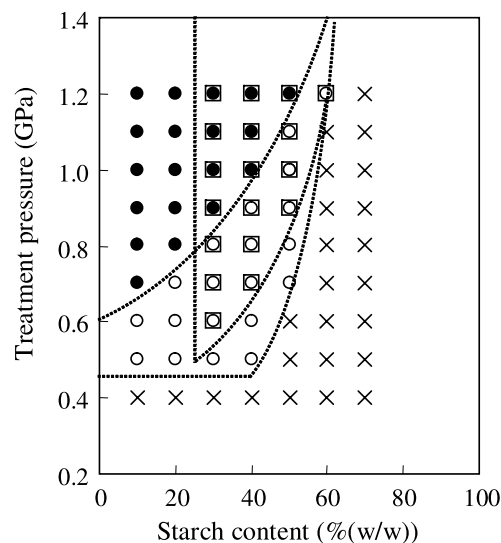


Fig. 5. State diagram of potato starch–water mixtures (treatment pressure vs. starch content). From the change in the ΔH_{gel} and ΔH_{retro} , the physically modified states of HHP-treated potato starch–water mixtures were classified into five states: cross, native state ($\Delta H_{\text{gel}} \geq 18$ J/g (dry potato starch basis) and $\Delta H_{\text{retro}} = 0$ J/g); open circle, partially gelatinized state ($\Delta H_{\text{gel}} < 18$ J/g and $\Delta H_{\text{retro}} = 0$ J/g); closed circle, gelatinized state ($\Delta H_{\text{gel}} = 0$ J/g and $\Delta H_{\text{retro}} = 0$ J/g); open circle embedded into square, partially gelatinized but retrograded state ($\Delta H_{\text{gel}} < 18$ J/g and $\Delta H_{\text{retro}} > 0$ J/g); closed circle embedded into square, retrograded state ($\Delta H_{\text{gel}} = 0$ J/g and $\Delta H_{\text{retro}} > 0$ J/g).

other botanical sources (Bauer et al., 2004; Błaszczak et al., 2005; Douzals et al., 1996; Hibi et al., 1993; Katopo et al., 2002; Muhr & Blanshard, 1982; Muhr et al., 1982; Selmi et al., 2000; Stute et al., 1996). This study demonstrated that the gelatinization pressure of potato starch–water mixtures increased with increased starch content, and 10–50% mixtures were gelatinized completely by HHP-treatment in the pressure range of 0.7–1.2 GPa. A part of our results is in agreement with a previous suggestion that the gelatinization pressure of 12.8% (w/w) potato starch suspension at ambient temperature was 0.8 GPa (Muhr & Blanshard, 1982). On the other hand, there is a study that will be inconsistent with a result of this study; Bauer and Knorr (2005) reported that 0.7 GPa was not sufficient to archive the complete gelatinization of 5% (w/w) potato starch suspension below 50 °C. This discrepancy of the experimental results might be ascribed to the difference in the methods of the determination of gelatinization. We used a calorimetric method, and Bauer and Knorr employed a microscopic method that counts loss of birefringence of at least 200 starch granules. Although the microscopic study is often used to determine the degree of gelatinization (Bauer & Knorr, 2004, 2005; Bauer et al., 2004; Douzals et al., 1996, 2001; Muhr & Blanshard, 1982), experimental results may depend strongly on the judgment by researchers. Although Douzals et al. (2001) demonstrated that a microscopic evaluation always overestimate the degree of gelatinization by 20% in comparison with a calorimetric one, Bauer and Knorr (2005) stated that the overestimation could not be

confirmed by their preliminary experiment. Muhr and Blanshard (1982) stated that heat-gelatinized potato starch still displayed weak birefringence. Similarly, we observed microscopically that very small granules of HHP-treated potato starch–water mixtures of $\Delta H_{\text{gel}} = \Delta H_{\text{retro}} = 0 \text{ J/g}$ sometimes kept the birefringence (the data not shown). From these facts, it is evident that the observation of gelatinization depends on the method of determination. Since it is beyond the scope of this paper to discuss a suitable analytical approach for the determination of gelatinization, it is noted that the interpretations in this study are based on the enthalpy change of gelatinization.

4.2. Retrogradation of potato starch–water mixtures by HHP-treatment

The DSC thermograms of 30–60% potato starch–water mixtures showed re-gelatinization of retrograded starch. Taking our experimental processes into consideration, it was estimated that the ΔH_{retro} value corresponded to the progress of retrogradation after a few hours of HHP-treatment. Retrogradation means recrystallization in gelatinized starch, and the progress depends strongly on the starch content of starch–water mixtures. According to a review on retrogradation of heat-gelatinized starch (Hoover, 1995), marked retrogradation was observed in the starch content range of 30–60% (w/w). In the starch content range, the degree of retrogradation increased with increased starch content (Miles, Morris, Orford, & Ring, 1985; Orford, Ring, Carroll, Miles, & Morris, 1987). These observations are reasonably in agreement with those of HHP-treated potato starch–water mixtures in this study; retrogradation was observed when ΔH_{gel} was below 12 J/g. This indicates that a certain amount of gelatinized part induced by HHP-treatment was required for the progress of retrogradation. Furthermore, ΔH_{retro} increased with increased potato starch content in the starch content range of 30–60%. This agrees just with the dependence of retrogradation on starch content of heat gelatinized starch–water mixtures (Hoover, 1995). In order to understand the mechanism of retrogradation of HHP-treated starch more in detail, further study on the processing time after HHP-treatment will be necessary.

4.3. State diagram of potato starch–water mixtures

Physically modified states of HHP-treated potato starch–water mixtures were summarized in Fig. 5. Some studies have presented time courses of the degree of gelatinization (Bauer & Knorr, 2004, 2005; Stolt et al., 2001) and/or pressure–temperature diagrams (Bauer & Knorr, 2005; Douzals et al., 2001; Rubens & Heremans, 2000) in order to summarize the physically modified states of HHP-treated starch. A state diagram of starch–water mixtures (treatment pressure vs. starch content), however, has never been reported. Physically modified states of HHP-treated potato starch–water mixtures will be overviewed by this state diagram. Five physical states are presented

from the changes in ΔH_{gel} and ΔH_{retro} : no change, partial gelatinization, complete gelatinization, partial gelatinization and retrogradation, and complete gelatinization and retrogradation. Since partial gelatinization and/or partial gelatinization and retrogradation can be required for resistant starch (Bauer & Knorr, 2005; Chung et al., 2006; Escarpa et al., 1996), combined use of HHP-treatment and the state diagram will enable the production of modified starches with controlled gelatinization and/or retrogradation.

Fig. 5 was resulted from HHP-treatment at 40 °C for 60 min, and thus, effects of treatment temperature and time were not investigated in this study. There will be little effect of treatment time on the HHP-gelatinization, because it has been reported that treatment time longer than 60 min shows a very small effect on the degree of gelatinization (Bauer & Knorr, 2004, 2005; Stolt et al., 2001). Treatment temperature, however, will affect the HHP-gelatinization significantly according to previous studies (Bauer & Knorr, 2005; Douzals et al., 2001; Muhr & Blanshard, 1982; Rubens & Heremans, 2000). On the other hand, effects of treatment temperature and time on the retrogradation are unclear at this stage. A foreseeable extension of this research would involve investigating the effects of treatment temperature and time on the physically modified states of HHP-treated potato starch–water mixtures and clarification of the mechanism of gelatinization and/or retrogradation induced by HHP-treatment.

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