



# Effect of high hydrostatic pressure treatment on conventional hydroxypropylation of maize starch



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## ABSTRACT

Effect of high hydrostatic pressure (HHP) treatment on conventional hydroxypropylation of maize starch was investigated. Three groups, 'Con' group (conventional hydroxypropylation), 'HHP-Con' group (HHP treatment before conventional hydroxypropylation), and 'Con-HHP' group (HHP treatment after conventional hydroxypropylation), were used in this work. The degree of substitution (DS) increased over the reaction time in all groups. Swelling power and solubility were high in HHP treated groups compared to Con group because HHP treatment weakens the binding forces inside starch granule. In the results of RVA, the Con-HHP group showed a lower pasting temperature and a higher breakdown and viscosity than the other groups. Pre- and post HHP-treatments altered the physicochemical properties of hydroxypropylated maize starch. Hydrophilic and bulky hydroxypropyl groups may weaken the bindings in the granule, while the HHP treatment promoted the collapse of granules and accelerated the leaching of intra-soluble materials.

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## 1. Introduction

Starches are widely employed as useful materials in the food industry. However, native starches do not have the physicochemical characteristics required for diverse processing conditions (heat treatment, acid/alkali conditions, shear stress, high flow velocity, etc.), so modified starches with improved pasting properties are mainly used in the industry. Among them, chemically modified starches are obtained by the addition of reaction reagents to the starch suspension and the induction of reactions for specific times at specific temperatures, leading to partial decomposition and derivatization processes. The physicochemical properties of the modified starches formed as such are affected by the kinds and properties of the added reagents (Bemiller, 1997; Jane, 1995; Singh, Kaur, & McCarthy, 2007). In addition, in the case of physical modification (another modifying method), specific conditions of temperature, time, and pressure are applied to a suspension of starches with a sufficient amount of water to cause granule structure collapse, distortion, or re-arrangement phenomena, leading to changes in the pasting properties (Bemiller, 1997; Gunaratne & Corke, 2007; Jayakody & Hoover, 2008).

Hydroxypropylated starch is a chemically modified starch obtained through etherification reactions between propylene oxide (PO) and the hydroxyl groups of carbons 2, 3, and 6 of anhydrous glucose in starch under alkali conditions (Chlebicki, 1975; Leegwater, Marsman, & Mackor, 1973). In this case, the substitution ratios of HO-2-carbon, HO-3-carbon, and HO-6-carbon are known to be 3, 3, and 6 (Wootton & Manatsathit, 1983). The substituted hydroxypropyl groups are bulky and hydrophilic, and weaken the binding force inside starch while increasing the binding force with water, thereby changing the properties and functionality of the granules (Gunaratne & Corke, 2007; Lawal, 2009). Through these changes, hydroxypropyl starches acquire improved freeze-thaw stability and cold storage qualities; increased swelling power, solubility, peak viscosity, and paste clarity; and reduced gelatinization temperatures and tendencies toward retrogradation (Bemiller, 1997; Chuenkamol, Puttanlek, Rungsardthong, & Uttapap, 2007; Liu, Ramsden, & Corke, 1999; Wootton & Manatsathit, 1983). However, hydroxypropylation reactions are problematic in terms of cost and efficiency, because these reactions require a long agitating process, amounting to 20–72 h.

Meanwhile, high hydrostatic pressure (HHP) treatment of starches has been attracting attention recently as a method of making physically modified starches, in that it leads to changes in granule structure and pasting properties (Jayakody & Hoover, 2008). It has been reported that hydration of non-crystalline regions (amorphous regions) occurs, followed by changes in the

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alignment of crystalline regions, leading to gelatinization when a certain level of pressure is applied, even without any specific temperature condition (Oh, Pinder, Hemar, Anema, & Wong, 2008). In addition, HHP treatment changes A-type starches into B-type starches (Hibi, Matsumoto, & Hagiwara, 1993; Katopo, Song, & Jane, 2002).

To overcome the limitations of conventional hydroxypropyl starches, Kim, Choi, Kim, and Baik (2011) conducted a study regarding hydroxypropylation using HHP, and reported that hydroxypropylation was initiated in 15 min. However, their study was limited to hydroxypropylation using only HHP, and did not sufficiently address the complex effects of HHP treatment and conventional chemically induced reactions. Therefore, in the present study, to investigate the effect of HHP on the conventional chemical modification process, modified starches were made from maize starch with conventional hydroxypropylation and HHP as pre- or post-treatments, and the physicochemical properties of the modified starches were examined.

## 2. Materials and methods

### 2.1. Materials

The maize starch with 10.5% moisture content (wet basis or w.b.) and 26.5% amylose content (dry basis or d.b.) was kindly donated from Daesang Corporation (Ichon, Korea). Propylene oxide (PO), anhydrous sodium sulfate and propylene glycol were purchased from Sigma-Aldrich (St. Louis, MO, USA).

### 2.2. Preparation of hydroxypropylated maize starch

To examine the effects of HHP on conventional hydroxypropylation, samples were prepared using the following three reaction treatments: conventional hydroxypropylation without HHP treatment (Con), HHP treatment before conventional hydroxypropylation (Con-HHP), and HHP treatment after conventional hydroxypropylation (HHP-Con). Conventional hydroxypropylation was carried out using the method of Wootton and Manatsathit (1983) with some modifications, and the HHP treatment was performed using the method proposed by Kim et al. (2011).

Starch suspension was made with native maize starch (20 g d.b.) and distilled water (80 ml), and anhydrous sodium sulfate ( $\text{Na}_2\text{SO}_4$ , 8%, starch basis [s.b.]) was added to the starch suspension with continuous stirring. The pH was adjusted to 11.5 using 1 M NaOH. Then, PO (12%, starch basis or s.b.) was added to the suspension, the suspension was transferred to a retortable pouch to be placed in the HHP container, and the pouch was hermetically sealed using a heat sealer. "Con" samples were incubated in a 45 °C water bath for 5, 10, 15, 20, and 24 h. "HHP-Con" samples were put into a HHP instrument (ISA-CIP-80-50-270; Ilshin Autoclave Co., Daejeon, Korea) and treated with a pressure of 400 MPa at room temperature for 15 min. HHP treated sample pouches were immediately transferred to a 45 °C water bath and incubated for 5, 10, 15, 20, and 24 h. "Con-HHP" samples were incubated in a 45 °C water bath for 5, 10, 15, 20, and 24 h. These sample pouches were immediately transferred to a HHP instrument (ISA-CIP-80-50-270; Ilshin Autoclave Co., Daejeon, Korea) and treated with a pressure of 400 MPa at room temperature for 15 min.

In all samples, after hydroxypropylation, pH was adjusted to 5.5 with 1 M HCl to terminate the hydroxypropylation reactions, and the samples were centrifuged at 1500g for 20 min and washed three times with distilled water. Samples were dried for 24 h at 40 °C, ground, and sieved using an 80-mesh sieve.

### 2.3. Degree of substitution (DS)

The hydroxypropyl contents of samples were measured using the method presented by Johnson (1969). Accurately weighed samples (50–100 mg, d.b.) were placed in 100 ml volumetric flasks, and 25 ml of 1 M  $\text{H}_2\text{SO}_4$  was added to each flask. Then, samples were hydrolyzed completely in boiling water and the sample solutions were left at room temperature to cool down. From each volumetric flask, 1 ml of the solution was transferred to a 25 ml test tube, and 8 ml of concentrated sulfuric acid (98%) was carefully added to the test tube and closed the test tubes. Reactions were induced for three minutes in boiling water, and the test tube was immersed immediately in ice water to cool down the sample. A 0.6-ml sample of ninhydrin reagent (3% ninhydrin in 5%  $\text{Na}_2\text{S}_2\text{O}_5$ ) was added to the test tube, and the test tube was left for 100 min in a 25 °C water bath. The sample solutions were diluted to 25 ml using concentrated sulfuric acid (98%) and the mixed well by slow shaking. An appropriate amount of the sample solution was transferred to a cuvette cell, and after five minutes for equilibration, the optical density was measured at a wavelength of 590 nm using a spectrophotometer (UV-1200, Labentech Co., Ltd., Incheon, Korea). Native maize starch was used as a control, and a standard curve was made using propylene glycol. The values measured through the spectrophotometer were multiplied by a conversion constant of 0.7763 and were used to calculate the degree of substitution using the following formula (Rutenberg & Solarek, 1984).

$$DS = \frac{162\text{Hydroxypropyl}\%}{5800-58\text{Hydroxypropyl}\%}$$

### 2.4. Paste clarity

The paste clarity of each sample was measured using the method of Craig, Maningat, Seib and Hoseney (1989). A 10% starch suspension was placed in a 15-ml tube and heated for 30 min in boiling water. At this time, the tube was shaken at five-minute intervals to prevent precipitation. After the sample was cooled down at room temperature, the transmittance (%T) value was measured at a wavelength of 650 nm using the UV-vis spectrophotometer.

### 2.5. Scanning electron microscopy (SEM) analysis

The granule morphologies of native, Con, HHP-Con and Con-HHP samples were examined using a scanning electron microscope (TM3000, Hitachi Ltd., Tokyo, Japan). For SEM, the starch samples were sprinkled on a double-sided carbon plate that was fixed on an aluminum stub. All the samples were examined using an accelerating voltage of 15 kV.

### 2.6. Swelling power and solubility

The swelling power and solubility of the samples were measured using the method of Koo et al. (2005) with some modifications. Each starch sample (0.5 g, d.b.) was mixed with distilled water (30 ml) and shaken for 30 min in a 90 °C water bath. After heat treatment, the sample was centrifuged at 3000g for one hour. The supernatant was dried at 105 °C, and the weights of the soluble starch and the precipitate were measured. Based on the measured weights, the swelling power and solubility were calculated using the following equations:

$$\text{Solubility}(\%) = \frac{\text{Weight of soluble starch(d.b.)} \times 100}{\text{Weight of starch(d.b.)}}$$

$$\text{Swelling power} = \frac{\text{Weight of precipitates(w.b.)} \times 100}{\text{Weight of starch(d.b.)} \times (100 - \text{solubility})}$$

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