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REVIEW

Characteristics of physically modified starches

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Abstract
Starch is an abundant natural, non-toxic, biodegradable polymer. Due to its low price, it is used for various purposes in various fields such as the cosmetic, paper, and construction industries as well as the food industry. Due to recent consumer interest in clean label materials, physically modified starch is attracting attention. Manufacturing methods of physically modified starch include pregelatinization, hydrothermal treatment such as heat moisture treatment and annealing, hydrostatic pressure treatment, ultrasonic treatment, milling, and freezing. In this study, toward development of clean label materials, manufacturing methods and characteristics of physically modified starches were discussed.

Keywords Starch · Physical modification · Clean label

Introduction

Starch is derived from the cereals, roots, tubers, and stems of various types of plants, and its physicochemical properties vary by source. Starch is composed of linear amylose and branched amylopectin as a combination of α-1,4 and α-1,6 linkages of glucose. The starch granules are composed of amylose and amylopectin, amylose is mostly a linear polymer with a molecular weight between 5×10^5 and 1×10^6, and amylopectin is a branched polymer with a molecular weight of several million (Belitz et al, 2008; Eliasson, 2006; Wang et al, 1998). The granules contain variably positioned amorphous and crystalline regions in the form of growth rings positioned within a lamellar structure (Gallant et al, 1997). Native starch is relatively dense and insoluble in water, limiting its use in the industry due to characteristics such as poor thermal stability. To solve this problem, starch is modified by physical, chemical, and enzymatic methods. However, consumers interest in clean label and physically modified starch is increasing because it can be classified as a component rather than an additive and can be used without constraints of regulations or labels. Additionally, physical treatments are generally easier to perform and often less expensive than chemical transformations and do not produce effluents containing unwanted reagents or reagent by-products.

In general, physical treatment of starch destroys or rearranges the packing structure of molecules in starch granules, resulting in various properties such as thermal stability and digestibility. Physical treatment is generally divided into thermal treatment and non-thermal treatment. The representative examples of thermal treatment are pregelatinization, heat moisture treatment, and annealing. Non-thermal treatment includes ultrasonic treatment, milling, freezing, and high hydrostatic pressure treatment. These various physical treatments can be combined depending on the intended outcome.

Thermal treatment

Pregelatinized starch

Pregelatinized starch is the most used physically modified starch. It has cold and hot water dispersibility, high viscosity, and is widely used for characteristics such as smooth texture. Since this starch has been dried to interrupt crystallinity, it swells immediately when put in water and quickly forms a viscose slurry. As such, it can be applied without additional

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heating. Due to these characteristics, this pregelatinized starch is widely used in food production.

There are three methods to produce pregelatinized starch: typical drum drying, extrusion, and spray drying (He et al, 2020; Majzoobi et al, 2011; von Borries-Medrano et al, 2018). Because these methods use different equipment and processing conditions, they result in starches with different characteristics. In the drum drying method, a starch–water slurry is quickly gelatinized and dried between counter-rotating drums heated with steam. The dried film is scraped from the drums and pulverized into various mesh sizes. The second process uses extrusion based on type of starch, moisture content, temperature, and screw speed. The characteristics vary depending on conditions. In this process, the extrudate is dried and ground into various sizes. The third spray drying method has the advantage of relatively less damage to the particles. The spray drying method depends on type of starch and moisture content, and powders with different characteristics can be obtained by controlling the inlet temperature and spray speed of the dryer (Fu et al, 2012).

Effects of temperature and processing time on production of pregelatinized starch using drum drying have been investigated (Karapantsios, 2006). In an investigation of the physicochemical properties of drum-dried pregelatinized wheat starch, the native starch granules were destroyed, the molecular structure was disrupted, and the crystallinity of starch was reduced. However, this type of starch shows cold water viscosity characteristics, high water absorption and expansion, and can be used as a thickener in various fields (Majzoobi et al, 2011). A study confirmed that pregelatinized starch was more soluble at room temperature than granular cold-water swollen starch (Li et al, 2014).

Various studies on extrusion manufacturing have been conducted, and differences in moisture content and solubility based on temperature and extrusion speed have been confirmed. Depolymerization may occur during the manufacture of pregelatinized starch, and the molecular weight of amylose and amylopectin of wheat starch decreased during the extrusion process (Colonna et al, 1984; Doublier et al, 1986). These changes were reported to be due to the high shear force generated in the extruder (Pei-Ling et al, 2010).

In the spray drying method, physicochemical properties such as thermal properties, solubility, and viscosity of pregelatinized starch vary by preheating temperature, starch concentration and heating time. A study using cassava starch confirmed that the viscosity characteristics, swelling power, and crystallization changed as the concentration and temperature increased (Santos et al, 2019). In a study using corn starch, changes in morphological characteristics of granule size and crystallinity were analyzed according to pretreatment temperature (Fu et al, 2012).

Chemically modified starches can also be used to make pregelatinized starch. In this case, a modified starch might be achieved after pregelatinization, but it may not be a pure physically modified starch. Pregelatinized starch products can be used in a variety of applications where heat is not available or where sufficient heat is not achieved for gelatinization and are added to improve texture and water retention in various types of food. Such starches can be useful as dry mixes for convenient home use. Because of its high water-holding capacity, it is used in products such as pudding, cream, and cake mix.

Granular cold-water-swelling (GCWS) starch

Granular cold-water-swelling (GCWS) starch is a type of pregelatinized starch, which is called instant starch, and it has viscosity and gel properties more similar to those of cook-up starches than previously mentioned pregelatinized starches (BeMiller and Huber, 2015). These starches are generally prepared in four ways. The first is heating amylose-containing starch in an alcoholic suspension (Eastman and Moore, 1984; Rajagopalan and Seib, 1991). The second is spray drying of the heated starch suspension, and the third is to treat the starch with an aqueous alkaline alcohol solution at room temperature (Chen and Jane, 1994b; Pitchon et al, 1981). The fourth method is instantaneous controlled pressure drop (DIC) (BeMiller and Huber, 2015). As GCWS starch can be prepared in various ways, several studies have been confirmed that property changes due to selection of various conditions in each process. In corn starch heated in an alcohol suspension state, decomposition of amylopectin molecules was confirmed with a decrease in crystallinity as viewed by X-ray diffraction pattern. In one study, the authors showed that amylopectin was decomposed during heat treatment and formed a complex similar to V-type amylose (Jane et al, 1986). The second method, spray drying, has been used for a long time, and differences in properties according to conditions have been confirmed (Pitchon et al, 1981). In addition, conditions of choice can be achieved by varying moisture content, pretreatment temperature, pH, drying method, and additives (Hedayati et al, 2016a, b; Wang et al, 2011; Zhu et al, 2019).

Use of an alkaline aqueous solution also can produce desired characteristics. In addition, starch treated under fixed conditions showed higher solubility in colder water. It has been reported that the solubility of corn starch decreases as the ethanol concentration increases (Chen and Jane, 1994a). Change also was verified by application of high pressure during ethanol treatment (Eastman and Moore, 1984). These methods cause only physical changes by modifying the basic structure of the D-glucopyranosyl unit.

Both pregelatinized starch (PG) and GCWS starch are used to achieve desired viscosity at room temperature, but they show important differences in functional properties due to molecular and granular differences. First, since PG is fully gelatinized, it does not show a gelatinized endothermic peak in thermal characterization, but it does maintain some microscopic birefringence indicating crystallinity (Hedayati et al, 2016a, b). Differences were also confirmed in morphological characteristics, with granules showing less damage (Yan and Zhengbiao, 2010). In addition, in a study conducted with buckwheat starch, GCWS showed relatively higher light transmittance and solubility than PG but lower crystallinity and swelling power (Li et al, 2014). Both processing methods can be used to achieve characteristics of a wide variety of end products. As there remain limitations to use of GCWS, it can be combined treatment with other physical non-thermal treatment technologies such as high hydrostatic pressure or ultrasonic treatments.

Heat moisture treated starch

Heat moisture treated (HMT) starch is heated at a temperature above the glass transition temperature (80–140 °C) for a specified period of time (1–24 h) at low moisture content ($< 35\%$). The first study in which HMT was performed to modify starch was reported in Sair and Fetzer (1944). HMT improves the internal structure by increasing the mobility of starch chains and spiral structures under limited moisture and high temperature conditions in order to achieve desired changes in the physical properties (Hoover, 2010; Hoover and Manuel, 1996; Jacobs and Delcour, 1998). HMT also destroys less stable structures allowing increased stability by changing or reorganizing internal structures (Luo et al, 2006). Before HMT, starch is sealed to retain moisture by preventing evaporation through use of high pressure. The energy of the excess water molecules is converted into kinetic energy that causes large-scale segmental movements and changes in the internal structure of starch (Wang et al, 2021). By inducing dramatic movement of water molecules through high heat treatment under limited moisture, different changes can be achieved under different conditions. In studies of the effect of HMT depending on the type of starch, it was reported that A-type crystalline starch was not modified and maintained its original polymorphic (Hoover, 2010; Zeng et al, 2015). Even in C-type crystalline starch, there was no change in pattern after HMT (Molavi et al, 2018). However, it has been reported that B-type crystalline starch can be converted into A-type or mixed form through HMT treatment (Perera et al, 1997; Varatharajan et al, 2011).

When HMT treatment was performed on various types of starch, gelatinization temperatures (onset temperature (T_o), peak temperature (T_p), and conclusion temperature (T_c)) generally increased and gelatinization enthalpy decreased (Pinto et al, 2015; Shin et al, 2005; Vasanthan et al, 1995; Vermeylen et al, 2006). Also, when HMT treatment is performed at a higher temperature, the size of the granules increases, viscosity characteristics decreases, and enthalpy decreases (Malumba et al, 2010). In most starches, swelling power and solubility decreased in HMT starch. As the water content and temperature increase during treatment, the solubility and swelling power decrease, which leads to increased crystallinity, crystallization transition from B-type to A-type, and formation of amylose–lipid complex. These changes were due to mutual or homogeneous binding of amylose and amylopectin, and the change in crystallinity pattern varies with temperature (Hoover, 2010; Varatharajan et al, 2011). In several studies on the viscosity characteristics of HMT starch, the pasting temperature and pasting time increased, and the peak viscosity and breakdown tended to decrease (BeMiller and Huber, 2015). The effects of HMT on setback or final viscosity differ by type of starch. It has been reported that properties such as stability of paste gels may vary depending on the type of starch (Gunaratne and Hoover, 2002; Hoover et al, 1993; Takaya et al, 2000).

In studies related to digestion characteristics, enzymatic degradation has been reported to decrease or show no change (Chung et al, 2009a, b; Englyst et al, 1992; Franco et al, 1995; Kweon et al, 2000). In addition, it was reported that the degree of digestibility could be affected by amylose or moisture content (Van Hung et al, 2016). Many studies on the digestion characteristics of HMT starch have been conducted and showed different tendencies based on type of starch. The mechanism of enzymatic reaction needs to be analyzed separately from the mechanism of thermal or viscosity characteristics.

Annealed (ANN) starch

Annealing is a hydrothermal treatment method of relatively long exposure to a temperature between the glass transition temperature (T_g) and the gelatinization onset temperature (T_o) under conditions of sufficient moisture ($> 40\%$). The achieved physical and chemical properties of annealing treatment are based on type of starch, temperature, and treatment time. It may be difficult to summarize the overall effect because numerous changes occur. Annealing treatment has been carried out for as short as 0.5 h (Kiseleva et al, 2004) and as long as 192 h (Gomes et al, 2004). The moisture content was also carried out under various conditions, up to 90% (Kohyama and Sasaki, 2006). Because of these various conditions, the results of general property changes are not as consistent as those of HMT starch. In general, annealing is performed above the glass transition temperature under sufficient moisture. These conditions increase the mobility of double helix chains inside starch granules to foster recrystallization, through processes such as aligning internal double helix structures (Gomand et al, 2012; Hoover, 2000; Vermeylen et al, 2006).

Previous studies have shown no significant changes in shape of starch granules with change in condition.

However, some reports noted reduced birefringence and increased porosity (Gough and Pybus, 1971; Rocha et al, 2012; Shi et al, 2021). In addition, some studies have reported change in crystalline pattern (Genkina et al, 2004), although it is more common for annealing to not affect the crystalline pattern (Chung et al, 2009a, b; Li et al, 2020; Rocha et al, 2011; Samarakoon et al, 2020). Generally recognized changes in thermal properties are increased gelatinization temperature and reduced temperature range of phase transition with increased annealing temperature (BeMiller and Huber, 2015; Jacobs and Delcour, 1998; Jayakody and Hoover, 2008; Liu, 2013). However, although the melting enthalpy of double helical structure (ΔH) generally is unchanged (BeMiller and Huber, 2015; Vermeylen et al, 2006; Wang et al, 1997), some studies have shown increased stability due to hydrothermal treatment (Hoover and Vasanthan, 1994; Nakazawa and Wang, 2003). Decreased stability that can be attributed to gelatinization of some weak crystalline phases (Tester et al, 1998; Vamadevan et al, 2013).

For solubility and swelling power, most studies show that annealing affects both solubility and swelling power (Jacobs and Delcour, 1998; Jayakody et al, 2007; Singh et al, 2011; Tester and Debon, 2000). In addition, the effect of annealing at 45 °C for 24 to 72 h was confirmed in three types of corn starch with different amylose and amylopectin contents. As the amylose content increased, the swelling power significantly increased, as did the annealing effect (Wang et al, 2014). The effect of annealing on viscosity properties is complex and differs by starch type. In general, annealing increases the pasting temperature and thermal stability and decreases peak viscosity and final viscosity (Adebowale and Lawal, 2002; Adebowale et al, 2005; Olu-Owolabi et al, 2011; Shih et al, 2007; Simsek et al, 2012; Song et al, 2014). Similar to the results of HMT, these results show a decrease in viscosity because swelling is limited due to structural stability inside the starch. On the other hand, some studies have reported higher peak viscosity after annealing (Jacobs et al, 1995).

Study on the digestibility of annealed starch was also conducted. Many studies have reported slight or no increase (Alvani et al, 2014; Chung et al, 2009a, b; Liu et al, 2015; Simsek et al, 2012). On the other hand, there have been some reports of a partial decrease in resistant starch. (Chung et al, 2009a, b; Song et al, 2014). In this way, annealing treatment of various types of starch under different moisture content, temperature, and time conditions shows irregular tendencies in various physicochemical properties. Therefore, in addition to moisture, temperature, and time conditions, more information about the properties of the starch itself are needed in the study of annealing treatment.

Non-thermal treatment

Ultrasonic treatment

Ultrasound waves have frequencies exceeding the threshold of human hearing. Ultrasonic treatment creates areas of intense heat and high shear stress. The detailed mechanism of sonication has been briefly described (BeMiller and Huber, 2015). Ultrasonic treatment of starch is performed under sufficient moisture and various characteristics and concentrations of dissolved gas, temperature, processing time, power, frequency, and amplitude. Many studies have investigated changes in starch properties based on the above factors. Depending on the intensity and duration of ultrasound treatment, partial gelatinization may occur due to the increase in temperature (Yu et al, 2013). In addition, in a study in which potato starch in suspension was subjected to ultrasonication, hydrogen gas produced conical pits in the granules, whereas air or oxygen gas produced surface erosion and holes, the size of which increased in inverse proportion to solubility. With carbon dioxide treatment, no damage was reported in the gas or vacuum conditions (Gallant et al, 1972). In addition to damage to granules, changes in swelling power, solubility, gel transparency, hardness, and adhesion have been identified (Jambrak et al, 2010; Majzoobi et al, 2014; Zheng et al, 2013).

In corn, potato, tapioca, and sweet potato starches, changes in viscosity were confirmed by ultrasonication. The viscosity of the starch solution was effectively reduced as the ultrasonication temperature increased (Iida et al, 2008). In addition, the influence of ultrasound was studied according to the amylose content in corn starch, and it had a greater influence on linear amylose than amylopectin. (Luo et al, 2008). In terms of thermal properties, ultrasonic treatment reduced the gelatinization enthalpy due to destruction of starch granules (Jambrak et al, 2010). Analysis of the viscosity characteristics of ultrasonicated starch near the onset of gelatinization showed decreased peak and final viscosities as well as particle size due to solubilization of starch aggregates by ultrasonication (Zuo et al, 2009). Ultrasonication damages the surface and weakens the granular structure and can cause a gelatinization-like reaction in some weak crystalline structures, which may result in other physical and chemical changes.

High hydrostatic pressure (HHP) treatment

High hydrostatic pressure (HHP) treatment is generally performed by subjecting the starch suspension to a pressure of 400 MPa or more. Adjusting the concentration, treatment time, and pressure of suspension for various types of starch allows a variety of gelatinization degrees to be obtained. When gelatinization is performed by HHP treatment, gelatinization is performed at various pressures depending on type of starch. When the temperature is increased, the required pressure may decrease for target gelatinization degree. Characteristic changes according to pressure, moisture content, and treatment time were observed in rice, as well as in various starches of barley, maize, and potato (Bauer et al, 2004; Błaszczak et al, 2005; Douzals et al, 2001; Katopo et al, 2002; Kim and Baik, 2022; Muhr and Blanshard, 1982; Stolt et al, 2000; Vallons and Arendt, 2009). A commonality of several studies is variation according to type of starch and increase in degree of gelatinization increased as temperature, pressure, moisture content, and treatment time increased.

HHP gelatinization proceeds with a different mechanism from heat-induced gelatinization, and various studies related to this difference have been conducted (Buckow et al, 2007; Douzals et al, 1996; Rubens and Heremans, 2000). In order to confirm differences in heat and pressure gelatinization mechanisms, changes in crystalline structure (XRD), short-range structure (FTIR), and lamellar and fractal structures (SAXS) have been confirmed (Liu et al, 2020; Yang et al, 2013). Gelatinization occurs when external water penetrates into the granule interior due to pressure, increasing the degree of hydration. During HHP treatment, the volume of starch suspension decreases and starch molecules are suspended in water. According to Le Chatelier's principle, hydration of the starch granules would be preferential because they occupy a smaller volume than the native suspension (Douzals et al, 1996, 1998). In addition, one of the differences from heat-induced gelatinization is that there is no agitation or mixing (shear force) during pressure treatment, which allows the process to proceed while maintaining the granules.

As there is a difference in gelatinization temperature according to type of starch, gelatinization pressure varies. Within the A-type crystalline structure, double helix packing results in a low water content and relatively high density, whereas the B-type crystalline structure is more open with a hydrated helical core. In support of this, many studies have reported that A-type starch is more sensitive to pressure than B-type starch. Wheat starch with an A-type pattern is most pressure sensitive. C-type (mixture of A and B types) tapioca starch showed intermediate properties, and B-type potato starch showed strong resistance to pressure (Bauer and Knorr, 2005). Potato starch generally reacts at 600 MPa or more, but it is possible to induce gelatinization at a lower pressure by increasing the water content (Kawai et al, 2007; Thevelein et al, 1981). The difference in sensitivity to pressure can be easily explained by the different packing arrangement of crystallites. However, even in the same type of starch, depending on the amylose content, the sensitivity to gelatinization pressure varies widely. In this regard, glutinous rice starch is more sensitive to pressure than normal rice starch (Oh et al, 2008).

There have been several reports on other physicochemical properties of starch modified by HHP. It was reported that in corn starch, gelatinization enthalpy decreased and gelatinization temperatures (onset temperature (To), peak temperature (Tp), and conclusion temperature (Tc)) increased when pressure of 500 MPa was applied, and gelatinization was complete at 600 MPa (Vallons and Arendt, 2009). Studies have reported that the solubility and swelling power of quinoa starch increase as the pressure (300–600 MPa) and temperature (25–70 °C) increase (Ahmed et al, 2018). However, in the case of mung bean starch, solubility and swelling power at 90 °C or higher decreased as the pressure increased (Li et al, 2011). In this way, HHP treatment under different conditions has various effects by type of starch. Compared to heat treatment of wheat starch, the gel obtained by pressure treatment showed fewer aging characteristics as almost no amylose leaching occurred due to maintenance of granular shape (Douzals et al, 1998). In addition, with taro starch, transparency, hardness, and adhesion are greater in pressure-treated gel than in that receiving heat treatment (Liu et al, 2013). There are also reports of increased gel strength in pressure treated gels due to some not fully gelatinized granules, resulting in increased G' and G" values (Guo et al, 2015). Pressure treatment in sorghum and buckwheat starch reduces viscosity characteristics such as peak viscosity and final viscosity because it limits the leaching of amylose and amylopectin (Ahmed and Al-Attar, 2017; Liu et al. 2016a, b, c). The physicochemical changes caused by HHP treatment of starch are attributed to limited leaching of amylose and amylopectin in the maintained the granules after gelatinization.

Similar physical modifications to those of conventional heat treatment methods (pregelatinized starch, HMT, ANN) can be induced by denaturation caused by pressure energy. Considering that water is an essential factor in pressure treatment as a transfer medium, pressure annealing as a new physical modification method can be performed under sufficient moisture conditions.

Freezing, thawing and freeze drying

When starch is frozen with water, fine ice crystals are generated while passing through the maximum ice crystal formation zone depending on the freezing speed. These crystals affect the structure of starch and associated features. During thawing, amylose or amylopectin are leached due to syneresis, producing change in chemical properties. The effect of freezing and thawing on potato starch confirmed particle surface damage and increased specific surface area.

(Szymońska and Wodnicka, 2005). In addition, the water matrix formed during deep freezing compresses the starch granules, which can cause leaching of amylopectin from the inside to the surface (Szymońska et al, 2000). It has also been reported that lyophilization weakens crystallinity in the internal structure of starch and damages the surface. This damage fosters enzyme access into the starch granules to increase digestibility (Apinan et al, 2007). However, in corn, where water transport is relatively limited, these changes were not observed (Larder et al, 2018; Zhang et al, 2014).

Methods for physical modification of starch are largely divided into thermal treatment and non-thermal treatment. Physical modification treatments have been applied to different types of starch under different conditions. Even in a specific type of starch treated under similar conditions, the changes in physicochemical properties were observed depending on the amylose and amylopectin contents. Thus, the outcomes of physical starch alteration might not be predictable. Because starch traits vary by type, detailed adjustments under various conditions such as temperature, moisture, and time will be required to affect the desired change. Most thermal treatment modification methods use water as a heat transfer medium to induce movement of water molecules and provide fluidity to the internal structure of starch, inducing restructuring. It might be possible to develop new physical modification methods with similar effects based on pressure rather than heat. In addition, a new physical process that is expected to have a synergistic effect will be able to proceed by continuously proceeding with the existing physical process of different mechanisms in two or three steps.

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Declarations

Conflict of interest None of the authors have conflicts of interest to disclose.

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