



# Thermal and rheological properties of tapioca starch gels with and without xanthan gum under cold storage



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## ABSTRACT

Changes in the thermal and rheological properties of tapioca starch (TS) with and without xanthan gum (Xan) (total polysaccharide concentration = 25% w/w) were investigated using a differential scanning calorimeter, Rheograph Gel and Texture Analyser. The gelatinization temperatures of TS shifted to higher values with the Xan concentration. Xan enhanced the retrogradation of TS during the initial stage of storage but retarded the process for a further storage time at 5 °C. The onset temperature of all reheated TS/Xan gels decreased with increasing storage time indicating thermally unstable structure formation after a longer storage time. Storage Young's moduli ( $E'$ ) of the TS and TS/Xan gels stored at 5 °C increased with increasing storage time. The  $E'$  values became more temperature dependent with storage time due to the weak cross-linkage of amylopectin molecules in the gels but became less dependent in the system containing Xan. TS/Xan gels kept for 14 days showed lower Young's moduli than TS gels from the compression test confirming retardation of the retrogradation process by Xan. The results suggested that Xan could retard the retrogradation of TS gels for longer storage times.

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## 1. Introduction

Starch composed of amylose and amylopectin has been widely used not only as a main ingredient in many products such as noodles and bakery products but also as a thickener, stabilizer, or gelling ingredient in food products. Starch used for food gel preparation plays an important role in product development especially for modifying the texture or shape setting. At high concentrations of starch, three-dimensional networks are obtained after starch dispersions are heated and cooled. Heating starch dispersions above a certain temperature leading to swelling and disintegration of starch granules is known as gelatinization. Then, upon cooling, starch gels are formed if the concentration of the starch paste is high enough (normally >6%) (Clark and Ross-Murphy, 1987; Miles et al., 1985) as a consequence of the aggregation of amylose chains forming ordered junction zones. The starch gelation process is also described as occurring after a hot paste containing amylose is cooled and becomes more elastic with solid-like characteristics (BeMiller, 2011). The starch gel, a thermodynamically unstable system, is generally regarded as composed of composite networks in which swollen starch granules are embedded in a continuous three-dimensional network of aggregated amylose

chains with junction zone formation (Ring, 1985) or entanglements called the initial phase of retrogradation. Further reorganization proceeds in starch gels during storage resulting in an increase in the rigidity of the gel by amylopectin (Biliaderis, 1991; Miles et al., 1985; Temsiripong et al., 2005; Yoshimura et al., 1996) depending on many factors such as the type of starch, the starch paste concentration, the heating and cooling conditions, the pH, and the presence of solutes such as salts and sugars (Swinkels, 1985).

Storage of starch-based gels with high moisture content at low temperature is one method to increase shelf life. However, this could enhance retrogradation in the products leading to changes in quality, especially in the texture of the stored product. Hydrocolloids have been widely used in food formulation to improve or to maintain the overall quality during distribution and storage by modifying the rheological and textural properties of foods. Many studies have reported the effect of hydrocolloids on starch paste and/or gel properties (Chen et al., 2009; Christianson et al., 1981; Kim and Yoo, 2006; Mandala and Bayas, 2004; Shi and BeMiller, 2002; Yoshimura et al., 1998). Tapioca starch (TS), produced from cassava roots, is differentiated from other starches because it contains about 17–20% amylose with very low protein and lipid contents (Breuninger et al., 2009). It is widely used in many products (Pongsawatmanit et al., 2007) because of the high viscosity, clear appearance, and low production cost, compared to other starches, especially in Southeast Asia (Biliaderis, 2009; Rapaille and Vanhemelrijck, 1997; Temsiripong et al., 2005). Xanthan gum

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(Xan) is a heteropolysaccharide produced by *Xanthomonas campestris* and provides excellent stability in thermal and acid systems. However, most reports on the thermal and rheological properties of the TS/Xan were investigated in a system with the total polysaccharide concentration at about 6% or lower (Chaisawang and Supphantharika, 2006; Chantaro and Pongsawatmanit, 2010a,b; Pongsawatmanit and Srijunthongsiri, 2008; Pongsawatmanit et al., 2011; Sikora et al., 2008). Sae-kang and Supphantharika (2006) investigated the thermal properties of a 24.0% TS/Xan mixture using differential scanning calorimetry (DSC) but the hardness and rupture strength were determined at 6.0% TS/Xan after repeated freeze–thaw cycles. For application in the food industry, both the thermal and rheological properties should be investigated at the same high total polysaccharide concentration to gain knowledge for further application in TS-based products such as pudding, jelly, and steamed layer dessert during manufacturing and storage.

Therefore, in this study, 25% w/w TS and TS/Xan systems were used to enhance the reassociation of starch molecules under low temperature storage. The effect of Xan on the gelatinization and retrogradation of the TS gel was evaluated using DSC. Changes in the rheological properties of cylindrical TS gels with and without Xan after storing at 5 °C were also investigated by observing the dynamic viscoelasticity and compression behaviors. The relationship between the thermal and rheological properties of TS and TS/Xan mixtures was established using the retrogradation parameter and Young's modulus. The results of this study are expected to be useful for TS-based product development in the food industry.

## 2. Materials and methods

### 2.1. Materials

Tapioca starch (TS) (Siam Modified Starch, Pathum Thani, Thailand) and xanthan gum (Xan) (Kelrol F, CP Kelco, San Diego, CA, USA) were used in the experiments. The moisture content of the TS and Xan was 11.1% and 8.6%, respectively, determined by a hot air oven method at 105 °C (AOAC, 2000). The amylose content of TS was 18.0% determined according to the method of Juliano (1971). The concentrations of both polysaccharides were calculated based on a dry basis and used without any further purification. Silicone oil with a viscosity of about  $0.65\text{--}1 \times 10^6$  mPa s was used as the heating medium to control the temperature and prevent water loss during measurement of the dynamic viscoelasticity.

### 2.2. Preparation of gels

Gel preparation and measurement procedures were performed according to Yoshimura et al. (1996) with a slight modification. Powders of TS or of the TS and Xan mixture were calculated and weighed for gel preparation of 25% w/w TS and TS/Xan (mixing ratio of 9.5/0.5 = 23.75% TS and 1.25% Xan, respectively). TS was dispersed in preweighed distilled water in a separable flask to achieve a total weight of 200 g using a motorized stirrer with a mixing blade (propeller shape with 40 mm diameter) at 340 rpm for 30 min at room temperature. In the case of the TS/Xan mixture, a precalculated amount of Xan was added to the preweighed distilled water and stirred for at least 6 h with a magnetic stirrer to ensure complete hydration of the polysaccharide before TS addition in a separable flask. The dispersion (TS or TS/Xan) was heated in an oil bath to 95 °C and heated further by holding at 95–98 °C for 30 min with continuous stirring at 340 rpm. Boiled hot distilled water was added into the hot mixtures for adjusting the total polysaccharide concentration to 25% w/w TS or TS/Xan dispersions and then mixed immediately. Hot mixtures were poured into cylindrical

Teflon molds with 20 mm diameter (20 mm or 30 mm height for the compression test and dynamic measurement, respectively), placed under a glass cover and packed in plastic bags before cooling in an ice-bath for 20 min. To prevent water evaporation, the gels were kept within the Teflon mold and covered with plastic film and finally packed in plastic bags before placing in a refrigerator at 5 °C. Samples were taken after 4, 7, 10, and 14 days for dynamic viscoelasticity measurement and a uniaxial compression test.

### 2.3. Determination of gelatinization and retrogradation of TS and TS/Xan mixtures using differential scanning calorimetry

The thermal properties of 25% w/w TS and TS/Xan mixtures with mixing ratios of 10/0, 9.875/0.125, 9.75/0.25 and 9.5/0.5 were measured using a differential scanning calorimeter (DSC822<sup>e</sup>, Mettler-Toledo GmbH, Greifensee, Switzerland). Precalculated amounts of Xan were added to preweighed distilled water and allowed to disperse throughout the sol for at least 6 h at room temperature using a magnetic stirrer to ensure full hydration of the polysaccharide. TS was added into the dispersions with continued stirring for a further 30 min. About 15 mg of the dispersions were weighed directly into a 40- $\mu$ L aluminum DSC pan and the pan was hermetically sealed. Gelatinization behaviors of TS/Xan mixtures were investigated by heating the pans from 25 °C to 110 °C at a heating rate of 5 °C/min. Another empty DSC pan was used as a reference. The onset temperature ( $T_0$ ), peak temperature ( $T_p$ ) and conclusion temperature ( $T_c$ ) were determined based on DSC thermograms. The sharpness of the transition peak was measured as the width at the half-peak height (the “half width”,  $\Delta T_{1/2}$ ). Gelatinization enthalpy ( $\Delta H_1$ ) expressed as J/g dry starch was evaluated based on the area of the main endothermic peak. After the first-run heating, the gelatinized TS and TS/Xan pastes were cooled down to 25 °C with a cooling rate of 10 °C/min and kept at 5 °C for 1, 4, 7, 14, 21, and 28 days. The stored samples were then reheated again to study the effect of Xan on the retrogradation of TS using the parameter of the retrogradation ratio determined by dividing the enthalpy of disintegration of the ordered structure ( $\Delta H_2$ ) in the second-run heating by the gelatinization enthalpy in the first-run heating ( $\Delta H_1$ ). Each sample pan was weighed before and after measurement to ensure that no weight was lost during the measurement. Four independent sets of samples were prepared and the average value was reported.

### 2.4. Dynamic viscoelasticity measurement of cylindrical TS/Xan gels kept under cold storage

The dynamic viscoelasticity of 25% w/w TS and TS/Xan (mixing ratio = 9.5/0.5) gels (cylindrical shape with 20 mm diameter and 30 mm height) were measured using a Rheograph Gel (Toyo Seiki Seisakusho Ltd., Tokyo, Japan). The storage ( $E'$ ) and loss ( $E''$ ) Young's moduli of TS and TS/Xan gels were determined as a function of the storage time according to Nishinari et al. (1980) and Yoshimura et al. (1996) with a slight modification. The temperature dependence of the  $E'$  and  $E''$  values for 25% w/w TS and TS/Xan gels stored at 5 °C for 4, 7, and 14 days were also measured by increasing the temperature of the gels from 20 °C to 55 °C (step ①), then decreasing from 55 °C to 20 °C (step ②), and finally increasing again from 20 °C to 55 °C (step ③). The  $E'$  and  $E''$  values were determined at 5 °C intervals. The measurement conditions were set within a linear viscoelastic regime as follows: longitudinal vibrations with frequency at 3 Hz, amplitude at 100  $\mu$ m and applied strain with a value of 0.0033. The measurement temperature was controlled in a silicon oil bath. Gels were held at each measured temperature for 15 min before starting the measurement process at each set temperature.

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