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Research article

Characteristics of texture and sucrose release after compression in tapioca starch gels containing sucrose and xanthan gum

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Abstract

Importance of the work: The texture of starch gels containing sucrose and hydrocolloids kept in cold storage plays an important role in sucrose release after compression.

Objectives: To characterize tapioca starch (TS) gels containing sucrose and xanthan gum (Xan) on texture and sucrose release after compression.

Materials & Methods: Mixtures were prepared of TS (25% weight per weight) and partial substitution of TS with Xan (0.125, 0.375 and 0.625%) containing sucrose (0–30%) to investigate their impact on retrogradation and the texture of the gels. The sucrose release of all TS/Xan/sucrose gels was determined using a repeated compression test at various speeds (20–500 mm/min) after keeping the gels at 5ºC for certain periods (1–14 d).

Results: Sucrose and Xan retarded the retrogradation of the stored TS gels. Sucrose addition enhanced the fracture stress (σ_f) and fracture strain (ε_f) in the TS gels. However, σ_f decreased and ε_f increased with increasing Xan content. Sucrose release (reported as total soluble solid after repeated compression) increased with increasing sucrose content and storage time but decreased with increasing compression speed. The number of fragments decreased with increasing sucrose and Xan contents and the compression speed based on image analysis. Sucrose and Xan revealed negative correlations with the retrogradation ratio (correlation coefficient, r = -0.455 and -0.398, respectively, $p < 0.01$) and positive correlations with fracture strain (r = 0.371 and 0.240, respectively, $p < 0.01$).

Main finding: The texture and sucrose release in selected starch gel could be modified using sucrose and Xan. The information gained could be applied in product development.

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Introduction

In the food industry, starch plays an important role in food gel product development, especially for improving the texture according to consumer requirements, with starch retrogradation (recrystallization) after gelatinization being the major factor contributing to the textural properties of starch-based food products (Kohyama and Nishinari, 1992). The addition of hydrocolloids to modify the texture and rheological properties of starch-based gels is widely used in the food industry to enhance the quality and shelf life of the starch gels (Shi and BeMiller, 2002; Funami, 2009; Pongsawatmanit et al., 2013). Hydrocolloids could control the release or absorption of various nutrients ranging from high molecular weight biopolymer, such as starches, to small molecules, such as vitamins, by modifying their network structure (Norton et al., 2014; Nishinari and Fang, 2016; McClements, 2021). In product development, sugar is one of the most important functional ingredients not only to enhance the sweetness and palatability of the product but also to modify food properties, such as rheological properties (Pongsawatmanit et al., 2007; Nishinari and Fang, 2016). With carbohydrates, smaller molecules can diffuse through viscous solutions or gel networks (Mills et al., 2011). Sucrose is the most common sweetener in the food industry with a low cost and provides many required functional properties in food products, such as moisture retention, texture and shelf-life extension, through its mechanical properties, water activity and hydrophilicity in food systems (Auerbach and Dedman, 2012; Jullapanya et al., 2021). Controlling the sucrose release or diffusion from the structure of processed foods is a challenge for food manufacturers to reduce the sugar consumption. Food gel texture influences the sucrose release as sucrose promotes the formation of hydrogen bonds and stabilizes the junction zone structure (Yang et al., 2015). Added sucrose increases the elastic modulus, fracture stress and fracture strain of polysaccharide gel systems (Wang et al., 2014; Yang et al., 2015; Kohyama et al., 2016). More brittle gels have a greater sweetness intensity (Morris, 1994; Wang et al., 2014; Nishinari and Fang, 2016) because of the larger total surface area when the gels are broken down. In addition, the sweetness intensity is enhanced by the new surface formation velocity in gelatin/agar gels because the perceived taste intensity is determined in a very short time at an early stage in oral processing (Sala and Stieger, 2013).

Modifications of the textural properties of polysaccharide gels using hydrocolloids and sucrose have been extensively investigated by many researchers, including for a tapioca starch (TS)-xyloglucan system (Pongsawatmanit et al., 2007), an agar system (Yang et al., 2015) and for xanthan gum (Xan), agarose and alginate (Russ et al., 2014). A starch/hydrocolloid/sucrose mixture is used to prepare starch-based products in food industry. Understanding its rheological properties during storage and sucrose release during consumption is important for food product development. However, most studies on sucrose release have been done using non-starch hydrocolloid gels. There have been no known published reports on the impact of ingredient release on the starch system, which plays an important role for application in food industry (especially in the starch gel system) regarding retrogradation parameters to sucrose release and compression speed, such as for binary composite gels of wheat flour and high amylose corn starch as a resistant starch (Mojarrad and Rafe, 2018). Therefore, the current study used TS/Xan/sucrose systems to investigate the characteristics of texture and sucrose release after compression in TS gel containing sucrose and Xan. The TS obtained from cassava roots is commonly used in food products as a thickener because of its low production cost (Pongsawatmanit et al., 2007). Xan is a heteropolysaccharide produced by *Xanthomonas campestris* and its viscosity can be maintained in the presence of salts over a wide pH range and at high temperature because of its excellent stability in thermal systems (Williams and Phillips, 2021). For these reasons, the objectives of the current study were to evaluate the effect of sucrose and Xan on the gelatinization and retrogradation of TS gels. A compression test was performed to evaluate the effect of sucrose concentration, storage time and compression speed on the sucrose release after fracturing from a compression test. The influence of the sucrose concentration and compression speed on the number of fragments was also examined using image analysis. The results gained from this study could be applied to further modification of the starch structure to provide lower sucrose addition but higher sweetness intensity in food product development using hydrocolloids.

Materials and Methods

Materials

Tapioca starch (TS) (amylose content = 21.7%) from Siam Modified Starch (Pathum Thani, Thailand) and xanthan gum (Xan) from CP Kelco (San Diego, CA, USA) were used without any further purification. The moisture contents of the

TS and Xan were 10.6% and 9.3%, respectively, on a wet basis, as determined using the hot-air oven method at 105°C for at least 6 hr until a constant weight was obtained (Association of Official Analytical Chemists, 2000). Sucrose (Sinopharm Chemical Reagent Co. Ltd.; Shanghai, China) was used as provided. Rhodamine B (Sinopharm Chemical Reagent Co. Ltd.; Shanghai, China) and sodium azide (Ajax Finechem Pty Ltd.; Taren Point, NSW, Australia) were of analytical grade.

Preparation of tapioca starch/xanthan gum/sucrose gels

Mixtures were prepared of 25% (w/w) TS and TS/Xan (mixing proportions of TS/Xan of 10/0, 9.95/0.05, 9.85/0.15 and 9.75/0.25) containing sucrose (0, 10, 20 and 30% w/w) with two replicates of each, according to the method of Pongsawatmanit et al. (2013), with slight modification. Xan dispersion was prepared by adding deionized distilled water and dispersing for at least 6 hr at room temperature (25±2°C). Then, the TS was added to the deionized distilled water or the Xan dispersion to obtain TS or TS/Xan dispersions, respectively. To ensure the complete hydration of polysaccharides, each dispersion was stirred using a magnetic stirrer for 2 hr at room temperature. Then the sucrose was added combined with continuous stirring for another 30 min. Each homogenous mixture was degassed for 15–40 min under vacuum, then heated to not more than 68°C for 2–4 min combined with stirring to achieve partial gelatinization of the starch granules (to avoid starch granule sedimentation during gel preparation) in a water bath (95°C). The hot dispersion (150 g) was transferred immediately into a plastic tube (250 mm length × 17 mm diameter), sealed and further heated at 95°C for 30 min. The gelatinized mixtures in the tubes were cooled immediately in an iced-water bath for 10 min prior to storage at 5°C before determination of their rheological properties. Separate 150 g samples (five for each preparation) were tested with one after each of 1 d, 2 d, 4 d, 8 d or 14 d of storage. One cylindrical gel for each treatment was cut into 18 cylindrical samples. The addition of sodium azide (0.02%) into each mixture was carried out to prevent microbial spoilage.

Gelatinization and retrogradation of tapioca starch/xanthan gum/sucrose mixtures using differential scanning calorimetry

The mixtures (30 g) of 25% (w/w) TS/Xan mixtures with and without sucrose were prepared according to the method described above without partial gelatinization and then the thermal properties were evaluated. Approximately 15 mg of each dispersion was weighed directly into a 40 µL aluminum differential scanning calorimeter (DSC) pan and was hermetically sealed. The gelatinization behavior of the TS/Xan/sucrose mixtures was determined by heating the pans from 25°C to 110°C at a rate of 5°C/min using a DSC (822^e; Mettler-Toledo GmbH; Schwerzenbach, Switzerland). A sealed empty pan was used as a reference. Gelatinization temperatures (at onset (T_o), peak (T_p) and conclusion (T_c)) were determined using heating thermograms. Gelatinization enthalpy (ΔH_1), expressed as joules per gram of dry starch, was investigated from the area of the main endothermic peak from the first-run heating. Then cooling the pans (at 10°C/min) to 25°C was carried out in the calorimeter prior to maintaining at 5°C for 1 d, 2 d, 4 d, 8 d or 14 d. After storage, the samples were reheated to investigate the influence of Xan and sucrose on the retrogradation of TS gels from the retrogradation ratio ($\Delta H_2/\Delta H_1$). The enthalpy of disintegration of the ordered structure (ΔH_2) was obtained from the second-run heating. Each sample pan was weighed before and after measurement to confirm that there had been no weight loss.

Compression test and analysis of released sucrose

The TS and TS/Xan cylindrical gels containing sucrose with different amounts were kept at 5°C for 1 d, 2 d, 4 d, 8 d or 14 d prior to sampling. At each time, one cylindrical gel for each treatment was cut into 18 cylindrical samples (17±1 mm diameter and 10±1 mm height). These gel samples were held at room temperature (25±2°C) for approximately 1.5 hr before measurement. Each gel sample was placed on an aluminum moisture can (55 mm diameter and 35 mm height) and 3 mL deionized distilled water was poured over it (Yang et al., 2015). Then a compression test was performed immediately at 25±2°C using a Texture Analyzer (TMS-Pro; Food Technology Co. Ltd.; Sterling, VA, USA) with a 2,500 N load cell linked to the Texture Lab Pro software program. Each gel sample was compressed to 1.5 mm (85% compression of the original sample height) for 10 compression cycles using a 50 mm diameter probe. Samples were compressed at different compression speeds (20 mm/min, 75 mm/min, 150 mm/min, 300 mm/min, 400 mm/min and 500 mm/min) according to Wang et al. (2014) and Yang et al. (2015), with a slight modification to the compression speeds. There were six measurements for each of the six speeds. Sucrose release was determined as total soluble solids (TSS) value after compression, using the same protocol. After an identical time (generally 3.5 min but 9 min for only the 20 mm/min compression speed) from the start of the test, a drop of the liquid was squeezed from a starch gel specimen after compression and collected using a plastic pipette. The TSS, presented as sucrose release, was measured using a refractometer (PAL-1; ATAGO Co. Ltd.; Tokyo, Japan), according to Wang et al. (2014) and Kohyama et al. (2016). The mechanical properties of the studied starch gels were evaluated based on large deformation (large strain >70% of the initial height) by the fracture stress and the properties appearing after fracture (Funami, 2011). The fracture stress and strain were reported after fracture of the gels; the barreling effect in the uniaxial compression tests was not considered.

An amount (3 mL) of water was added according to the preliminary test instead of 0.8 g (Yang, et al., 2015) in the agar gel system because of the reabsorption of exuded liquid into the starch gel. This was intended to mimic the addition of saliva. The quantity of saliva secretion during oral processing has been reported to be approximately 0.3 mL/min (Dawes et al., 2015; Werfalli et al., 2021). However, in the current study, the roles of digestive enzyme amylase and other ingredients in human saliva were not taken into account because the introduction of these substances would have complicated the problem; consequently, only water was added in the repeated compression of gels as a first approximation.

The first peak in the force-distance curve corresponding to initial gel fracture was used to calculate the fracture stress (σ_f) and fracture strain (ε_f) of the cylindrical gels, as described in Yang et al. (2015) and shown in Equations 1 and 2, respectively:

$$\sigma_f = F/A_1 = F/(\pi R_1^2) = Fh_1/(\pi R_0^2 h_0) \tag{1}$$

$$\varepsilon_f = |\ln h_1/h_0| \tag{2}$$

where F is the force (N) at fracture, A_1 (m^2), R_1 (m) and h_1 (m) denote the cross-section area, the radius and the height of the gel at fracture, respectively, R_0 (m) and h_0 (m) are the initial radius and the height of the cylindrical gel, respectively. In large deformation experiments, Hencky strain is commonly used, as shown in Equation 2.

Image analysis of tapioca starch/xanthan gum/sucrose gel fragments produced by fracturing

After keeping the TS and TS/Xan gels containing the different amounts of sucrose at 5°C for 14 d, the gels were cut into 10 mm length cylinders (17±1 mm diameter and 10±1 mm height) and held at room temperature (25±2°C) for 1.5 hr. Then, the gel was soaked in 0.1% Rhodamin B (Sinopharm Chemical Reagent Co. Ltd.; Shanghai, China) for 1 min before placing in an aluminum moisture can (55 mm diameter and 35 mm height). Deionized distilled water (3 mL) was added around the gel. Gel specimens were fractured using compression (85% compression) with 10 compression cycles and at different compression speeds (20 mm/min, 75 mm/min, 150 mm/min, 300 mm/min, 400 mm/min and 500 mm/min). Following fracture, all gel fragments were manually separated as a single layer on a filter paper and placed on a metal sieve using a spatula. The fragments on the filter paper were dehydrated at 25±2°C for 20–22 hr before scanning using the CamScanner application on a mobile phone (iPhone6; Apple Inc.; Cupertino, CA, USA). The number of fragments was analyzed using the free ImageJ, software program (INTSIG Information Co., Ltd.; Shanghai, China), modified from Yang et al. (2015).

Statistical analysis

The measurements were carried out using at least two freshly prepared samples and reported as the mean value ± SD. The experimental data were analyzed using analysis of variance with the SPSS V.12 statistical software package (SPSS (Thailand) Co., Ltd; Bangkok, Thailand). Significant differences ($p < 0.05$) were tested using Duncan's multiple range test. The characteristics of texture, sucrose release (as TSS) and the retrogradation ratio were compared based on Pearson's correlation coefficient (r) values.

Results and Discussion

Gelatinization and retrogradation of tapioca starch and tapioca starch/xanthan gum gels with and without sucrose

Gelatinization is a process that occurs when starch granules are heated in water and the crystalline region in the granules is changed into an amorphous region. A differential scanning calorimeter was used to determine starch gelatinization and the extent of starch retrogradation, where a large endothermic peak was observed (data not shown) from the first-run heating process for each mixture of 25% w/w TS/Xan (10/0, 9.95/0.05, 9.85/0.15 and 9.75/0.25) with and without sucrose, indicating the breakdown of the native starch structure (Pongsawatmanit et al., 2013). The gelatinization temperatures of the TS and TS/Xan mixtures in the absence of sucrose were in the range 54.5–82.6°C, which was under the endothermic peaks, suggesting the disruption of the clusters of amylopectin chains (Taggart and Mitchell, 2009). However, all TS/Xan mixtures containing sucrose had the higher values of T_o, T_p and T_c for all mixtures with sucrose addition compared to those without sucrose with ranges of 58.1±0.3–73.3±0.2°C, 73.3±0.5–89.4±0.3°C and 86.1±0.2–102.1±0.2°C (Table 1) for 10%, 20% and 30% sucrose addition, respectively, as was expected from the increase in the effective concentration of TS and the reduction in available water for the gelatinization process because of the sucrose-starch interaction. The addition of Xan shifted the gelatinization temperatures of TS to slightly higher temperatures (Table 1). A good agreement was also observed in a system of 25% w/w TS and xyloglucan with sucrose addition (Pongsawatmanit et al., 2007).

The gelatinization enthalpy (ΔH_1) is the energy required to disintegrate starch granules and is evaluated using the area under the large endothermic DSC curve from the first-run heating. The ΔH_1 values for the two options of the only TS and selected TS/Xan (9.75/0.25) dispersions containing 0–30% sucrose were approximately 16.4–17.9 and 15.0–17.1 J/g dry starch ($p < 0.05$, Table 1), respectively. The retrogradation was evaluated using the second-run heating endothermic peak originating from the disintegration of reordered structure formed during storage at 5°C (Kohyama and Nishinari, 1991; Pongsawatmanit et al., 2013). A smaller endothermic peak was observed on reheating than that of the first-run heating (data not shown); however, the area under the curve increased with a longer storage time of the gels at 5°C, suggesting a higher association of starch molecules during cold storage at 5°C. In the current study, the retrogradation ratio [(ΔH_2)/(ΔH_1)] was used to show the retrogradation degree (Kohyama and Nishinari, 1992) in stored TS gel samples with and without sucrose addition as a function of storage time (Fig. 1).

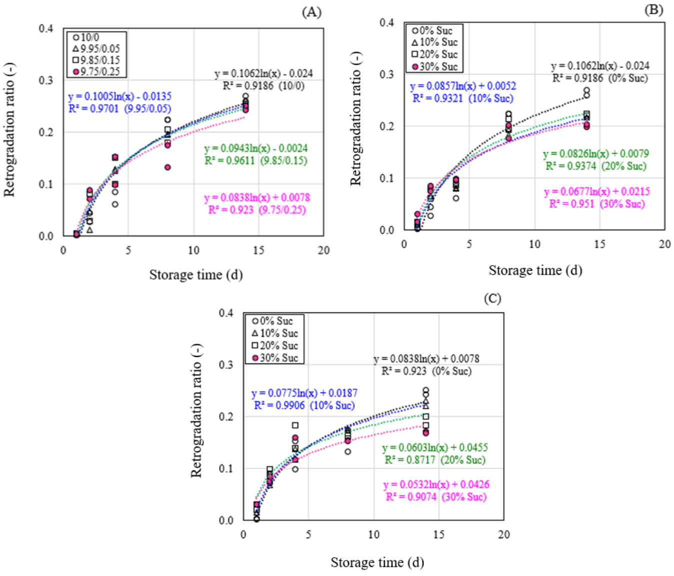

Fig. 1 Retrogradation ratio based on gelatinization enthalpy ($\Delta H_2/\Delta H_1$) of: (A) 25% (w/w) tapioca starch (TS)/xanthan gum (Xan) mixtures (10/0, 9.95/0.05, 9.85/0.15 and 9.75/0.25) without sucrose; (B) 25% w/w TS containing sucrose (0, 10, 20 and 30%); (C) TS/Xan gum (9.75/0.25) containing sucrose (0, 10, 20 and 30%), where data are presented as a function of storage time at 5°C, using DSC reheating rate of 5°C /min (n = 2), with all data plotted to show the spread of the data in the distribution and R^2 = coefficient of determination

Table 1 Effect of sucrose on DSC gelatinization parameters of 25% (w/w) tapioca starch (TS)/xanthan gum (Xan) mixtures (10/0, 9.95/0.05, 9.85/0.15 and 9.75/0.25) at heating rate of 5°C/min

Sucrose (%)	TS/Xan mixture	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g dry starch)
0	10/0	$55.3±0.2^j$	$69.1±0.3^o$	$82.0±0.3^j$	$16.4±1.1^{bc}$
	9.95/0.05	$54.5±0.5^l$	$69.8±0.2^n$	$81.9±0.3^j$	$15.1±0.7^{de}$
	9.85/0.15	$54.9±0.5^k$	$70.2±0.2^m$	$82.3±0.2^i$	$14.2±0.7^f$
	9.75/0.25	$55.8±0.2^i$	$70.5±0.1^l$	$82.6±0.2^h$	$15.0±0.9^e$
10	10/0	$58.1±0.3^h$	$73.3±0.5^k$	$86.1±0.2^g$	$16.7±0.7^b$
	9.95/0.05	$58.2±0.2^h$	$73.7±0.2^j$	$86.3±0.3^g$	$15.5±0.3^{de}$
	9.85/0.15	$58.6±0.1^g$	$74.2±0.2^i$	$86.6±0.1^f$	$15.3±0.5^{de}$
	9.75/0.25	$59.0±0.3^f$	$74.4±0.2^h$	$86.5±0.2^{df}$	$15.5±0.3^{de}$
20	10/0	$64.8±0.1^e$	$78.8±0.3^g$	$92.4±0.4^e$	$17.0±0.7^b$
	9.95/0.05	$64.8±0.1^e$	$79.8±0.2^f$	$92.3±0.1^e$	$15.8±1.0^{cd}$
	9.85/0.15	$64.8±0.5^e$	$80.4±0.2^e$	$92.8±0.2^d$	$15.8±0.6^{cd}$
	9.75/0.25	$65.7±0.2^d$	$80.8±0.1^d$	$93.2±0.3^c$	$15.8±1.4^{cd}$
30	10/0	$72.1±0.1^c$	$87.1±0.2^c$	$101.6±0.1^b$	$17.9±0.4^a$
	9.95/0.05	$72.3±0.2^c$	$88.5±0.3^b$	$101.5±0.3^b$	$17.1±0.5^b$
	9.85/0.15	$72.7±0.2^b$	$89.3±0.5^a$	$101.9±0.1^a$	$16.6±0.8^b$
	9.75/0.25	$73.3±0.2^a$	$89.4±0.3^a$	$102.1±0.2^a$	$17.1±0.4^b$

T_o = onset temperature; (T_p) = peak temperature; (T_c) = conclusion temperature; ΔH = gelatinization enthalpy.

Mean ± SD values (n = 10) in each column superscripted with different lowercase letters are significantly ($p < 0.05$) different.

The retrogradation ratio increased clearly with increasing storage time and was smaller for the TS and TS/Xan gels with higher sucrose contents than for gels with lower sucrose contents, indicating that the higher sucrose content in the gels contributed to greater retardation in the retrogradation process. For the short storage time (1–4 d), the effect of sucrose on the retrogradation ratio was not so dependent on sucrose concentration, while it was strongly dependent on the concentration of sucrose for samples stored for 14 d, since the retrogradation consisted of two separable processes. The first stage was governed by the gelation of amylose solubilized during gelatinization and the second stage was induced by the recrystallization of amylopectin within the gelatinized granules (Miles et al., 1985). For the storage times of 8 d and 14 d, the retrogradation decreased in the TS gels containing Xan (Fig. 1C). A lower retrogradation rate was observed in the TS/Xan gels than for TS alone, suggesting retardation of the amylopectin reassociation by Xan during cold storage. These results were in agreement with other reports involving replacement of starch in small quantities by glucomannan (Yoshimura et al., 1996) or xyloglucan (Temsiripong et al., 2005) enhanced the retrogradation process in the initial storage stage but could retard an increase in the retrogradation ratio of the starch gels in the later stage.

Fracture properties of tapioca starch gels with and without Xan and sucrose

The 25% (w/w) TS/Xan cylindrical gels (17 mm diameter and 10 mm height) with and without sucrose were prepared to determine any changes in compression behavior. The σ_f and ε_f values of the TS/Xan gels stored at 5°C for 1–4 d were not calculated because the gel was not broken under the compression at 85% deformation. However, the gels were deformed under all tested compression speeds. Only the TS/Xan gel (9.75/0.25) containing 30% sucrose could not be evaluated due to the limitation of available water to prepare for complete hydration of both TS/Xan and sucrose. Therefore, no data of the gel characteristics for this option were obtained. The σ_f and ε_f values of samples could not be calculated from the higher compression speeds (400 and 500 mm/min) because the gel was not broken. The σ_f and ε_f values of the gels kept for 8 d and 14 d were evaluated and presented based on the values obtained from the 75 mm/min compression speed because this was the lowest compression speed for identical times of 3.5 min prior to the collection of the exuded liquid from the gels.

The σ_f values of the TS gels increased with increasing sucrose concentration but decreased with increasing Xan content for both storage times (8 d and 14 d), as shown in Figs. 2A and 2C, respectively. However, the value of ε_f increased with both increasing sucrose and Xan concentrations (Figs. 2B, 2D). A firmer and more brittle appearance was observed for the TS gels than for the TS/Xan after keeping for 14 d at 5°C. There was better agreement for the higher σ_f and lower ε_f values of the TS gel than for the TS/Xan gel from the compression test. These results indicated that sucrose enhanced the strength of the gel structure and modified the starch gels to a higher deformable material, as observed with the increase in both the σ_f and ε_f values. Sucrose addition (10–30%) influenced the effective starch concentration in the systems, leading to an increase in both the σ_f and ε_f values for the TS and TS/Xan gels, as was expected from an increase in the elastically active network chains formed by sugar. Sucrose addition enhanced both the σ_f and ε_f values, which was in good agreement with other studies of agar gel systems (Wang et al., 2014; Yang et al., 2015; Kohyama et al., 2016) and gellan gels (Kawai et al., 2007; Evageliou et al., 2010), suggesting that sucrose enhanced the strength of the starch gel network. Furthermore, the substitution of a part of TS with Xan retards the retrogradation of TS gels from the association of the amylose and amylopectin molecules. The σ_f values of the TS/Xan gels increased at a slower rate than for the TS gels stored for a longer time at 5°C (Fig. 2).

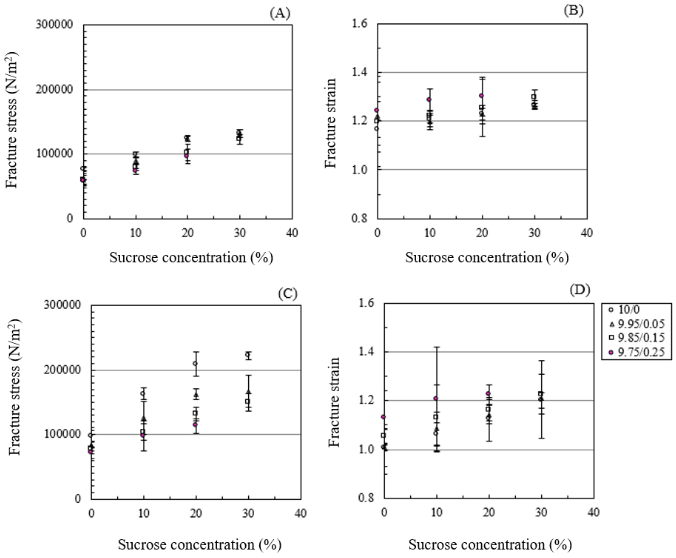

Fig. 2 Fracture stress (A, C) and fracture strain (B, D) of 25% (w/w) tapioca starch (TS)/xanthan gum (Xan) gels (10/0, 9.95/0.05, 9.85/0.15 and 9.75/0.25) kept at 5°C for 8 d (A, B) and 14 d (C, D) as a function of sucrose concentration, where gel compressed to 85% deformation at 75 mm/min compression speed using a 50 mm diameter probe at 25±2°C ($n = 6$), with no data for fracture stress and fracture strain from TS/Xan gel (9.75/0.25) containing 30% sucrose due to limitation of water available for hydration of both polysaccharides and sucrose during gel preparation and error bars indicate ± SD

The σ_f values of the TS/Xan gel stored for 14 d were lower than those for the TS gels (hard and brittle) but the ε_f values of TS/Xan (soft and deformable) were higher than for the TS gels.

Sucrose release of tapioca starch and tapioca starch/xanthan gum gels

After 3 mL deionized distilled water was poured over the cylindrical TS gels kept at 5°C for 4 d, 8 d and 14 d, a compression test (85% compression) for 10 compression cycles was performed immediately. Liquid was exuded after 3.5 min from starting the compression test of the starch gel from all compression speeds but after 9 min for only the 20 mm/min compression speed from the preliminary test to measure TSS. The TSS could not be used as a sucrose release ratio because exuded liquid was reabsorbed into the starch gel when the plunger was raised. However, in Fig. 3, only the 75 mm/min compression speed was used to present the TSS as a function of sucrose concentration for each storage time.

The TSS of the exuded liquid from the starch gels is used to indicate the release of sucrose. In fact, sucrose could be released from the starch gel to the surrounding water by free diffusion (Wang et al., 2014). However, faster release was observed in the breakage or fragmentation. The TSS as a function of sucrose concentration revealed higher values with increasing sucrose concentration due to the higher sucrose concentrations in the systems (Fig. 3). Considering the storage time (4 d, 8 d, 14 d) at 5°C, the TSS from the exuded liquid increased with increasing storage time. The TSS increased with increasing Xan during the initial stage of storage (Fig. 3A) but decreased for longer storage times (8 d and 14 d, Figs 3B and 3C, respectively), with these result being related with retrogradation ratio of the TS and TS/Xan gels (from Fig. 1) suggesting that Xan retarded sucrose release in the TS gel for longer storage times.

The TSS values in the exuded liquid at 25% w/w for all TS/Xan/sucrose gels were plotted as a function of compression speeds after storage at 5°C for 14 d following compression for 10 cycles, as shown in Fig. 4. Increasing the compression speed decreased the TSS of the starch gels. The low compression speeds at 20 mm/min and 75 mm/min produced high TSS values (high sucrose release) from each gel, especially from the gels containing a higher sucrose content (Fig. 4), due to the breakage of the gels under the first compression.

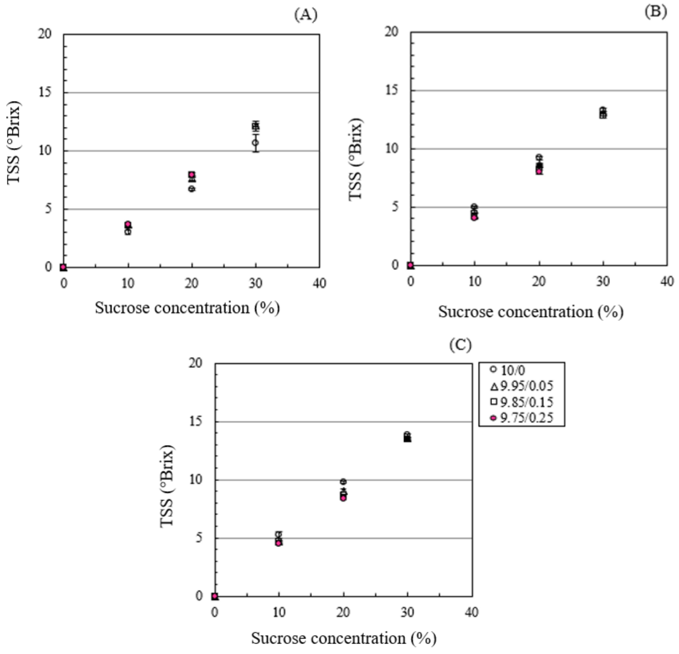

Fig. 3 Total soluble solids (TSS) of exuded liquid as function of sucrose concentration of 25% (w/w) tapioca starch (TS)/xanthan gum (Xan) gels (10/0, 9.95/0.05, 9.85/0.15 and 9.75/0.25) kept at 5°C for: (A) 4 d; (B) 8 d; (C) 14 d, where liquid collected after 3.5 min from start of compression, gel compressed to 85% deformation at 75 mm/min compression speed for 10 cycles using a 50 mm diameter probe at 25±2°C (n = 6), no data for TSS from TS/Xan gel (9.75/0.25) containing 30% sucrose due to limitation of water available for hydration of both polysaccharides and sucrose during preparation and error bars indicate ± SD

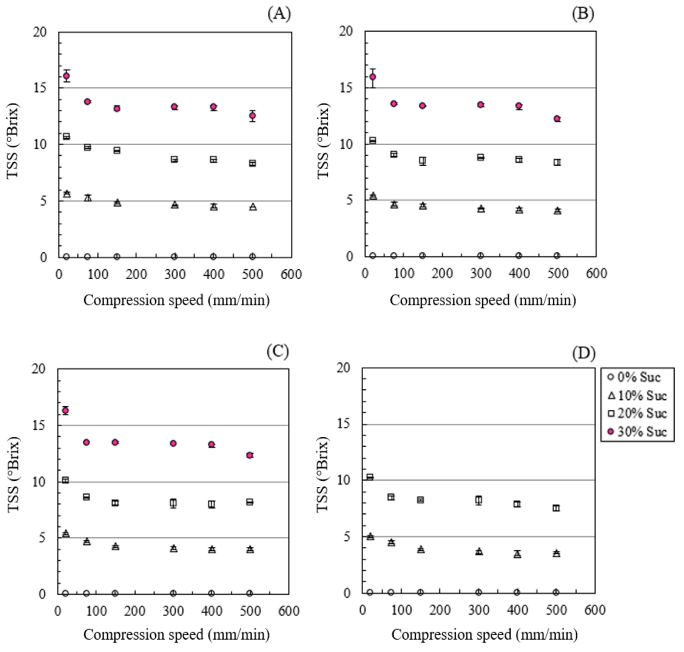

Fig. 4 Total soluble solids (TSS) of exuded liquid as function of compression speed of 25% (w/w) tapioca starch (TS)/xanthan gum (Xan) gels at mixtures of (A): 10/0; (B) 9.95/0.05; (C) 9.85/0.15; (D) 9.75/0.25, where sample contain sucrose (Suc, 0–30%) and stored at 5°C for 14 d, gel was compressed at 85% for 10 cycles using a 50 mm diameter probe at 25±2°C (n = 6), no data for TSS from TS/Xan gel (9.75/0.25) containing 30% sucrose (Suc) due to limitation of water available for hydration of both polysaccharides and sucrose during preparation and error bars indicate ± SD

Number of fragments of tapioca starch/xanthan gum/sucrose gels

After 10 repeated compression cycles of the TS and TS/Xan gels containing different sucrose contents kept for 14 d at 5°C with different speeds of compression, the number of fragments was determined and plotted as a function of compression speed (Fig. 5). Sucrose addition decreased the number of fragments from the repeated compression test. The results show good agreement with the food gels of agar-gelatin-sucrose systems (Mosca et al., 2015) and agar gels (Yang et al., 2015). However, low compression speeds (20 mm/min and 75 mm/min) enhanced the number of fragments, leading to a faster release of sucrose (higher TSS), as shown in Fig. 4 from the starch gels. The number of fragments of the TS/Xan gels decreased with increasing compression speed (between 20 and 500 mm/min). The results showed good agreement with other studies, such as a system of 2% agar containing 20% sucrose (Wang et al., 2014) and gellan gels (Nakamura et al., 2001). Yang et al. (2015) reported that the number of fragments of agar gels decreased with increasing sucrose contents due to an increment in the fracture stress.

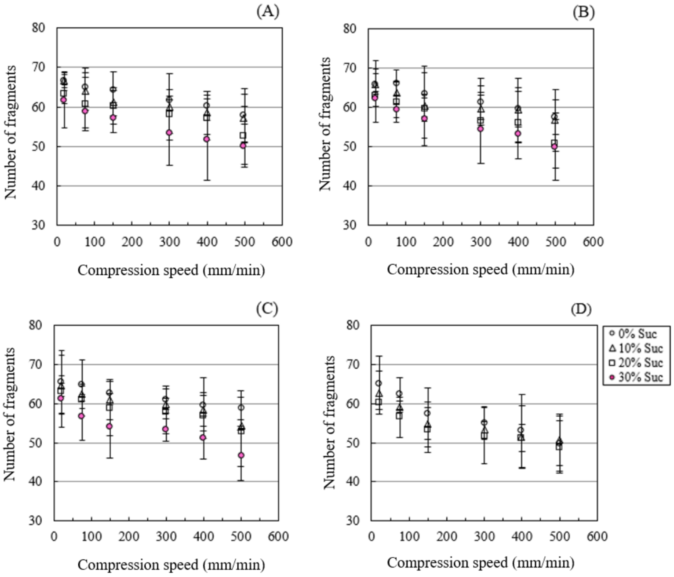

Fig. 5 Number of fragments as a function of compression speed of 25% (w/w) tapioca starch (TS)/xanthan gum (Xan) gels at mixing proportions of: (A) 10/0; (B) 9.95/0.05; (C) 9.85/0.15; (D) 9.75/0.25, where samples contain a range of sucrose (Suc, 0–30%) and stored at 5°C for 14 d, gel was compressed to 85% for 10 cycles using a 50 mm diameter probe at 25±2°C (*n* = 4), no data for total soluble solids from TS/Xan gel (9.75/0.25) containing 30% sucrose due to limitation of water available for hydration of both polysaccharides and sucrose during preparation and error bars indicate ± SD

The number of fragments was plotted as a function of the retrogradation ratio of the TS/Xan/sucrose gels after storage for 14 d (Fig. 6A, $R^2 = 0.768$). A linear master curve of the number of fragments was obtained. A higher number of fragments of the starch gels depended on the degree of retrogradation, indicating that the TS and TS/Xan gels under cold storage had a higher degree, consistent with a hard and brittle gel. However, there was a negative linear relationship between the number of fragments and fracture strain (Fig. 6B, $R^2 = 0.902$).

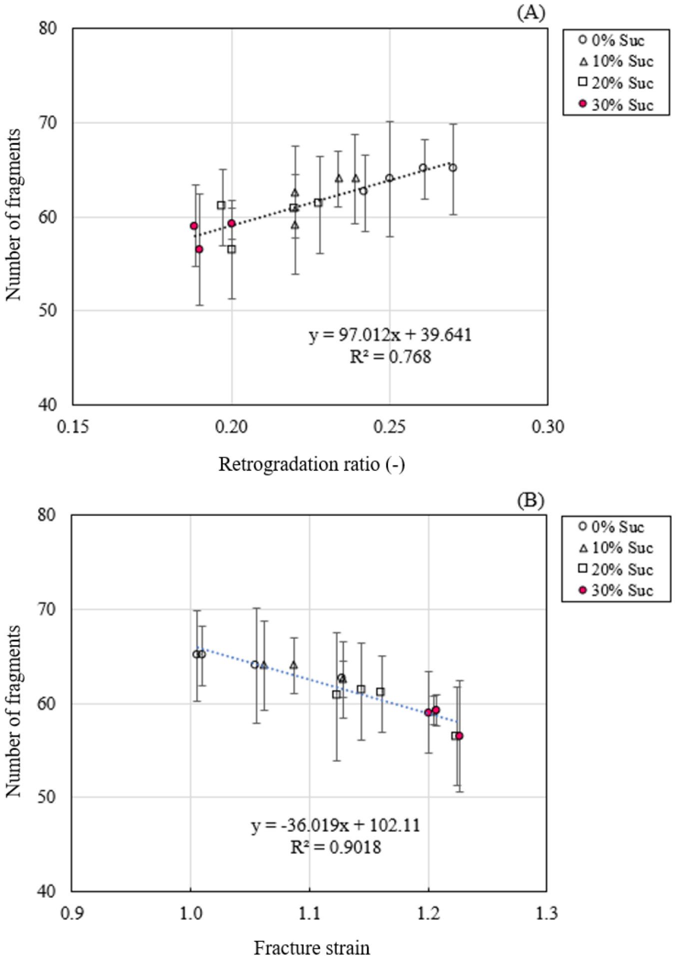

Fig. 6 Relationship of 25% (w/w) tapioca starch/xanthan gum gels containing sucrose (Suc, 0–30%) after storage at 5°C for 14 d: (A) number of fragments and retrogradation ratio; (B) number of fragments and fracture strain, where number of fragments based on gel compression at 85%, 75 mm compression speed for 10 cycles with a 50 mm diameter probe at 25±2°C, R^2 = coefficient of determination and error bars indicate ± SD

Correlation analysis

All correlations among the TSS, fracture stress, fracture strain, number of fragments and retrogradation ratio of the TS and TS/Xan gels with and without sucrose were evaluated, as shown in Table 2. Pearson's correlation coefficient (r) was used to quantify the relationship between Xan, sucrose, storage time, TSS, fracture stress, fracture strain, number of fragments and the retrogradation ratio of TS/Xan gels (10/0, 9.95/0.05, 9.85/0.15 and 9.75/0.25). Xan had a negative correlation with the TSS, fracture stress and retrogradation ratio (r = -0.195, -0.454 and -0.398, respectively, $p < 0.01$) but a positive correlation with fracture strain ($r = 0.240$, $p < 0.01$), confirming that the retrogradation process in the TS gels could be retarded by using Xan. Sucrose had significant, strong, positive correlations with TSS ($r = 0.996$, $p < 0.01$), fracture stress ($r = 0.706$, $p < 0.01$), and fracture strain ($r = 0.371$, $p < 0.01$). However, sucrose had a negative correlation with the number of fragments ($r = -0.401$, $p < 0.01$) and retrogradation ratio ($r = -0.455$, $p < 0.01$) suggesting that sucrose retarded the retrogradation, leading to lower the number of fragments, as shown in Fig. 6A. Storage time had a significant effect on the correlation of texture parameters, as shown in Table 2. Positive correlations were obtained between storage time and fracture stress, number of fragments and retrogradation ratio (r = 0.432, 0.490 and 0.627, respectively, $p < 0.01$); however, storage time had a negative correlation with fracture strain ($r = -0.480$, $p < 0.01$). TSS has a positive correlation with fracture stress ($r = 0.746$, $p < 0.01$) and fracture strain ($r = 0.344$, $p < 0.01$) whereas it had a negative correlation with the number of fragments ($r = -0.378$, $p < 0.01$) and retrogradation ratio ($r = -0.349$, $p < 0.01$). These results confirmed that the sucrose release (presented as TSS) could be related to the textural properties of the TS/Xan gels and retrogradation during cold storage.

In conclusion, sucrose addition increased the gelatinization temperatures of all the TS/Xan mixtures but retarded the retrogradation of the TS gels. Xan enhanced the retrogradation and the sucrose release of the TS gels in the initial storage stage at 5°C but decreased the retrogradation and sucrose release of the TS gels after longer storage. With increasing sucrose concentration, the σ_f and ε_f values increased, while Xan decreased the values for σ_f but increased the values for ε_f in the concentration range examined. The number of fragments of TS gel decreased with increasing sucrose, Xan content and compression speed. Therefore, for short shelf-life products (about 1–4 d), Xan addition to enhance the retrogradation process in this initial storage stage could be used for higher sucrose release. The information gained in this study could be further applied in TS-based food products kept in cold storage in terms of product texture and sucrose-containing food.

Conflict of Interest

The authors declare that there are no conflicts of interest.

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Table 2 Correlation matrix between total soluble solids (TSS), fracture stress, fracture strain, number of fragments at 75 mm/min, compression speed and retrogradation ratio at different sucrose concentrations (0–30%) and storage times (8 d and 14 d) of 25% (w/w) tapioca starch gels with and without xanthan (Xan)

	Xan (%)	Sucrose (%)	Storage time (d)	TSS (°Brix)	Fracture stress (N/m^2)	Fracture strain (-)	Number of fragments	Retrogradation ratio (-)
Xan (%)	1.000							
Sucrose (%)	0.000	1.000						
Storage time (day)	0.000	0.000	1.000					
TSS (°Brix)	-0.195**	0.996**	0.034	1.000				
Fracture stress (N/m^2)	-0.454**	0.706**	0.432**	0.746**	1.000			
Fracture strain (-)	0.240**	0.371**	-0.480**	0.344**	-0.172*	1.000		
Number of fragments	-0.150	-0.401**	0.490**	-0.378**	-0.028	-0.440**	1.000	
Retrogradation ratio (-)	-0.398**	-0.455**	0.627**	-0.349**	0.128	-0.701**	0.562**	1.000

* = correlation significant at $p < 0.05$ (2-tailed);

** = correlation significant at $p < 0.01$ (2-tailed);

TSS, Fracture stress, Fracture strain ($n = 6$), Number of fragments ($n = 4$), Retrogradation ratio ($n = 2$)

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