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Effect of dry heat treatment with xanthan on waxy rice starch

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ABSTRACT

Waxy rice starch was impregnated with xanthan and heat-treated in a dry state. The effects on the pasting and rheological properties of the treated starch–xanthan mixture were evaluated. Swelling of the granule was restricted, and a continuous rise of the viscosity during pasting was provided for the treated sample. After pasting, the gel forming ability of the treated starch was strengthened, as both storage and loss modulus increased and tan δ decreased. The paste also owned the highest zero order Newtonian viscosity and yield stress. An increase in starch particle size of the dry heated starch–xanthan mixture suggested a cross linking of the starch granules by the xanthan polymers. An increase of crystallinity was observed for the starch after dry heat treatment, but with the addition of xanthan the amorphous region of the granule became more resistant to dry-heating. The melting enthalpy was found to be correlated with the crystallinity.

1. Introduction

Starch is an important ingredient in many food and industrial products. To increase its functionality as an additive in the products, starch can be modified in several ways. Most commercial starches employed in either processed food or industrial applications are chemically modified to improve their physicochemical properties (Raina, Singh, Bawa, & Saxena, 2006). However, from safety considerations, processes to produce modified starches that use little or no chemical agents and produce less waste are preferred.

Heat treatment of starch changes its granular and molecular structure, and the changes depend on the presence of moisture (Chung et al., 2007). Heating starches under dry conditions is a method to produce modified starches. With respect to other chemical methods, dry heating is a simple, safe method and produces little if any byproducts. Chiu et al. (1998) introduced a dry heating process for formulating physically modified starches. They reported that thermally treated starches have been considered to be functionally equivalent to chemically cross-linked starches (Chiu et al., 1999).

Besides heat treatment, different types of gums (hydrocolloids) that are mixed with the starch have been reported to improve the characteristics of starch products (Rosell, Yokoyama, & Shoemaker, 2011; Sikora, Kowalski, & Tomasik, 2008; Tischer, Noseda, de Freitas, Sierakowski, & Duarte, 2006; Sudhakar, Singhal, & Kulkarni, 1992). Lim, Han, Lim, and BeMiller (2002) first reported the research on modification of starch by dry heating with ionic gums. It was found that dry heating with sodium alginate or carboxymethylcellulose (CMC) raised the paste viscosity of waxy maize starch, but reduced that of potato starch. As compared to sodium alginate and CMC, a dry heat treatment with xanthan provided the most substantial changes in paste viscosity of the starches (Lim, BeMiller, & Lim, 2003). Chung et al. (2007) found that the waxy rice starch heated with the mixture of phosphate salts and xanthan exhibited a continuous increase in pasting viscosity. In our previous study, rice starches with different amylose content were heat-treated in a dry state after being dispersed with low or medium viscosity CMC. The modified waxy starch showed the most significant change in viscosity throughout pasting, implying that the interaction were mostly occurred between the hydroxyl groups in amylopectin of rice starch and carboxylate acid groups of CMC (Li, Shoemaker, Ma, & Shen Zhong, 2008). To study the reaction mechanism between the starch and ionic gum through dry heating, more studies are needed.

The cooking procedures for many starch products and their cooked characteristics are related to starch gelatinization and pasting properties, and the starch pastes formed after gelatinization is useful in many food applications for desirable rheological properties. By dry heat treatment with an ionic gum, the modified starch may have different rheological properties after pasting. In this study, waxy rice starch was dispersed with xanthan and heat

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treated in a dry state. The pasting and rheological properties of the heated dispersion were evaluated. Additional studies on the granular and molecular structure of the starches were also carried out to obtain more information about the effect of dry heat treatment with an ionic gum on the starch.

2. Materials and methods

2.1. Materials

Waxy rice (CM101) was grown and milled by the California Cooperative Rice Research Foundation, Biggs, CA. Protease N 'Amano' was provided by Amano Pharmaceutical Co., Ltd. (Nagoya, Japan). Xanthan was obtained from Sigma Chemical Co. (St. Louis, MO, USA).

2.2. Isolation of rice starch

Waxy rice starch (protein content 96%, dry basis) was isolated from waxy CM101 rice (Li, Shoemaker, Ma, Luo, & Zhong, 2009). Milled rice (50 g) was soaked in 150 mL of deionized water for 18 h. The rice and water were then blended in a blender for 3 min. The initial pH of the solution was adjusted to 8.5 using 1.0 M NaOH, and Protease N (100 units/mL) was added. The protease hydrolysis was conducted at 50 °C for 4 h with constant stirring at 160 rpm. After the hydrolysis, the slurry was centrifuged at 10,000 × *g* for 10 min. The supernatant and the surface brown layer of the starch were removed and the lower white starch layer was washed with deionized water, followed by centrifugation. The washed starch was freeze–dried, passed through a 200 mesh sieve and stored at room temperature until used.

2.3. Starch modification by dry heat treatment with xanthan

The starches were dry heat treated with xanthan according to Lim et al. (2002) with some modifications. Xanthan (0.3 g) was dispersed in distilled water (40 mL) with vigorous stirring at 250 rpm. Waxy rice starch (9.7 g, dry basis) was added to the gum dispersion and stirred for 60 min at room temperature. The whole dispersion was transferred into a glass dish and dried at 40 °C in an oven to the moisture content of less than 10% through gravimetrically determination and grounded into powder. The starch–xanthan powder was then heated in an electric oven at 130 °C for 4 h (SX-H). Heated (S-H) and unheated (S-U) waxy rice starch dispersions (10 g/40 g water) were prepared and dried as controls. Dispersions of unheated starch–xanthan mixture (SX-U) were also prepared without the dry heating step.

2.4. Pasting properties

The pasting properties of the native or modified starch samples were measured in triplicate on a rotational rheometer (AR G2, TA instrument, New Castle. DE, USA) as described by Li, Shoemaker, Ma, Ibanez-Carranza, & Shen Zhong, 2008) and Zhong, Li, Luo, Ma, & Shoemaker, 2009. A cone and plate geometry was used. The cone made from a polysulfoneplastic, had a 4° angle and was 40 mm in diameter. The native and modified starch dispersions (8.8%) (w/w, dry weight basis) were prepared, and a calibrated pipette was used to deliver 1.1 mL of a dispersion between the cone and plate, which was the exact volume to fill the gap. A thin layer of silicon oil was dispensed around the perimeter to minimize evaporation during heating.

The following program was set up: equilibration at 50 °C for 1 min, a linear temperature increase to 95 °C at a rate of 12 °C/min, holding at 95 °C for 2 min 30 s, a cooling step with a linear temperature decrease to 50 °C at the same rate and holding at 50 °C for 1 min. The shear rate during the measurement was maintained at 200 s^{-1}, so that sedimentation would be prevented for the starch granules before swelling and pasting at this rate.

2.5. Rheological properties

Immediately after the completion of the pasting program, the temperature was raised to 65 °C, and the sample was held for 5 min at rest before continuing. In the next step, the viscoelastic properties of the pastes were measured at 65 °C, according to Li et al. (2009). An oscillatory stress sweep was made at a constant frequency of 1 Hz over an oscillatory stress range of 0.1–10 Pa at 5 points per log cycle. The storage (G') and loss modulus (G'') were determined in triplicate from measurements within the identified linear viscoelastic range of each paste.

The measurement of viscosity under a controlled shear stress range was made in triplicate following the oscillatory measurements at 65 °C according to Zhong et al. (2009). Different shear stress ranges were used for each sample, based on an observed apparent yield value of each paste, which had been observed in initial measurements. For the measurement of a flow curve, measurements were made at a series of shear stresses, starting with the lowest value. At each shear stress the paste viscosity was observed until either an equilibrium value was obtained or at the end of 2 min. The final value of viscosity was recorded and the next shear stress was applied.

2.6. Particle size distribution

Native or modified starch samples (100 mg) were dispersed in 5 mL of water and stirred at 200 rpm for 10 min. The dispersion was then swiftly transferred into the sample cell of the particle size analyzer (Laser S3500, Microtrac, Montgomeryville, PA, USA). The flow rate of water was set at 60 mL/s, and run for 30 s. Each sample was analyzed in triplicate.

2.7. X-ray diffraction

The X-ray diffraction patterns were made with the sample powders (10% moisture), using a diffractometer (BrukerD8 AXS, Bruker BioSpin, Rheinstetten, Germany) in duplicate at target voltage 40 kV and target current 100 mA with 0.154 nm CuK radiation (Ni filter). The typical widths of the divergence, scattering and receiving slits are 1.0, 1.0 and 0.2 mm, respectively. Diffractograms were recorded from 3° to 55° with a step of 0.5°. Relative crystallinity of the starches was calculated according to the method of Nara, Sakakura, and Komiya, 1983 using peak-fitting software (Origin 7.0, Microcal Inc., Northampton, MA).

2.8. Thermal properties

Thermal properties of the samples were analyzed with a differential scanning calorimetry (DSC) (Q2000, TA instrument, New Castle. DE, USA). An aluminum pan without anything was used as a reference. The sample (3 mg, dry weight basis) was weighed precisely into aluminum sample pan and mixed with water to obtain a ratio of 1:2 (w/w). The pans were hermetically sealed and stored at room temperature for 15 h. After loading on the DSC, the pans were then held at 25 °C for 1 min, heated from 25 to 140 °C at10 °C /min and cooled to 25 °C at the same rate.

Table 1
Pasting curve data of waxy rice starch (S) and waxy rice starch–xanthan mixture (SX) either heated (-H) or unheated (-U) in a dry state.

Samples	Pasting onset temperature (°C)	Peak temperature (°C)	Peak viscosity (Pa s)	Final viscosity (Pa s)
S-U	66.6 ± 0.1^{c}	73.9 ± 0.3^{ab}	0.74 ± 0.05^{b}	0.71 ± 0.03^{b}
S-H	64.1 ± 0.8^{a}	73.3 ± 0.4^{a}	0.67 ± 0.04^{a}	0.60 ± 0.02^{a}
SX-U	66.6 ± 0.2^{c}	72.6 ± 0.6^{a}	1.04 ± 0.06^{c}	0.84 ± 0.07^{c}
SX-H	65.8 ± 0.1^{b}	–	–	0.91 ± 0.09^{d}

Values are means ± SD of triplicate.
Values in the same column with different superscripts are significantly different ($p \leq 0.05$).

2.9. Statistical analysis

The statistical analysis of the results was conducted by the analysis of variance (ANOVA; SAS Statistic Package; SAS, Cary, NC, USA). Significant differences were defined as $P \leq 0.05$.

3. Results and discussion

3.1. Pasting properties

The pasting characteristics of waxy rice starch (S) and starch–xanthan (SX) mixture before (-U) or after dry heat treatment (-H) were measured (Table 1). Pasting onset temperature reflects the first measurable swelling of the starch granules as reflected by the appearance of an increasing viscosity. It was found that the onset temperatures of S-H and SX-H decreased with dry heat treatment. Dry heating would break intra- or inter-molecular hydrogen bonds of starches, which would facilitate the swelling of starch granules and result in the decrease of pasting onset temperature. Pasting peak temperature reflects the beginning of granule distortion or disruption when the granular structure cannot support continued swelling. No signigicant difference was observed for the peak temperature among the samples (Table 1).

In addition to the disruption of hydrogen bonds by dry heating, the excessive heat could have cleaved glycosidic linkages, which could have decreased the viscosity. Dry heat treatment without xanthan significantly reduced the peak and final viscosities of the waxy rice starch (Fig. 1). Similar results have been reported by Lim et al. (2002). Rice starch–xanthan mixture without dry heating (SX-U) showed the highest peak viscosity. After dry heat treatment, there was no peak viscosity of SX-H. The viscosity continued to increase during the holding period at 95 °C, and the final viscosity was the highest among all the treated and untreated samples (Fig. 1). The starch granule seemed to be wrapped by the xanthan molecules through multiple points of connection. Thus, the swelling of the starch granule was restricted and the disruption of the granule was completely prevented even at the maintaining period of the temperature at 95 °C. It also suggested that shear-stabilization of the starch granule was provided by dry heat treatment with xanthan, which was similar to those modified starches obtained with chemical cross-linking. Previous reports suggested that ester bond could be formed when the starch and ionic gum mixture were dry heated (Lim et al., 2003; Li et al., 2008a).

3.2. Rheological properties

Immediately following the pasting program on the rotational rheometer, the temperature on the paste was changed to 65 °C, and the storage (G') and loss (G'') modulus were recorded within the linear viscoelastic range of each paste. Steeneken (1989) used dynamic measurements as a function of time to report that rheological measurements of starch paste at 60 °C could be made with no retrogradation effects. In this study, 65 °C was selected to limit the degree of further retrogradtion effects after the pasting curve program was completed.

Dynamic rheological spectra of the native and modified rice starches showed that the elastic modulus was always higher than the loss modulus, and there was no crossover of G' and G'' observed between the two moduli for any of the samples throughout the frequency range of measurements (data not shown); these gels could be classified as weak gels (Achayuthakan & Suphantharika, 2008; Viturawong, Achayuthakan, & Suphantharika, 2008; Clark & Ross-Murphy, 1987). The addition of xanthan for either unheated (SX-U) or heated (SX-H) waxy rice starch increased G' and G'' of

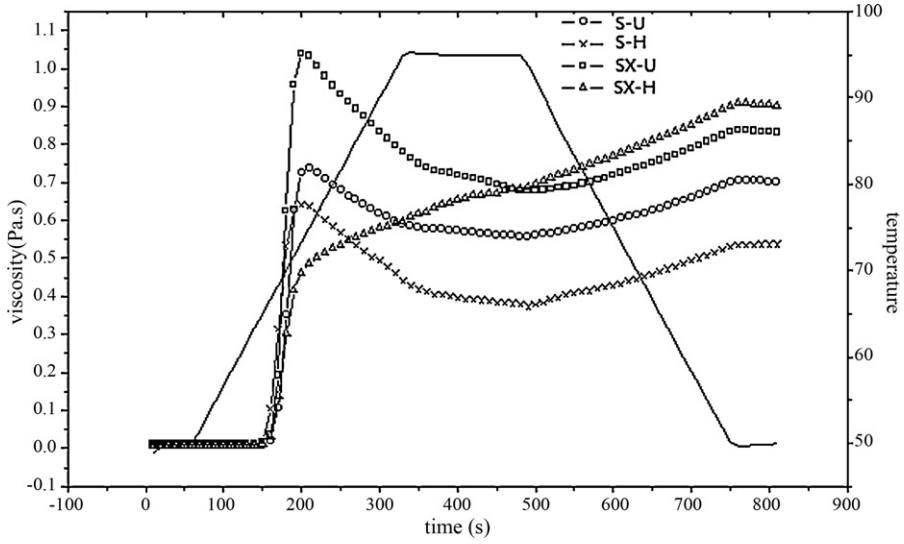

Fig. 1. Waxy rice starch (S) and waxy rice starch–xanthan mixture (SX) either heated (-H) or unheated (-U) in a dry state.

Table 2
Rheological parameters for the pastes of waxy rice starch (S) and waxy rice starch–xanthan mixture (SX) either heated (-H) or unheated (-U) in a dry state.

Samples	Storage modulus G' (Pa)	Loss modulus G''(Pa)	Loss tan δ	$\lvert G^*\rvert$ (Pa)	Zero-order Newtonian Viscosity η_o (kPa s)	Yield stress σ_o (Pa)
S-U	51.6 ± 1.6^a	10.6 ± 0.4^a	0.21 ± 0.001^b	52.7 ± 1.6^a	52.8 ± 3.2^a	21.5 ± 0^a
S-H	48.4 ± 3.1^a	10.7 ± 0.2^a	0.22 ± 0.011^b	49.6 ± 3.1^a	–	–
SX-U	67.0 ± 3.1^b	14.1 ± 0.6^b	0.21 ± 0.002^b	68.5 ± 3.0^b	93.2 ± 4.5^b	29.3 ± 0.3^b
SX-H	157.6 ± 10.7^c	24.1 ± 0.4^c	0.15 ± 0.008^a	159.5 ± 10.7^c	223.6 ± 6.1^c	54.0 ± 0.5^c

Values are means ± SD of triplicate.
Values in the same column with different superscripts are significantly different ($p \leq 0.05$).

both pastes (Table 2). While the G' value of SX-H was 2.3 times of the SX-U paste, and tan δ value of the SX-H paste decreased to 0.15, indicaing a higher degree of elasticity. The $|G^*|$ for the SX-H dramatically increased approximately 3.0 times of the native starch (S-U) and 2.3 times of SX-U. $|G^*|$ represents the magnitude of the compulex modulus, which is the overall strength of the gel. Consequently, the gel forming ability of the waxy rice starch was best strengthened after the dry heat treatment with xanthan.

The flow curves of the hot pastes (65 °C) were measured immediately after the oscillatory test. This was a measurement of viscosity as a function of shear stress. The steady state flow curve of the pastes showed an apparent yield stress and shear-thinning behavior (Fig. 2). The viscosities were constant at low shear stresses for each paste, which was called a zero-order Newtonian region, and followed by a shear-thinning region. The transition between the Newtonian and shear-thinning region was separated by a rapid fall of viscosity of several orders of magnitude over a narrow range of shear stress. The shear stress at this transition has been interpreted as an "apparent yield stress" (Barnes, 2000). The paste of SX-H had the highest yield stress (σ_o) and Newtonian viscosity (η_o). Similar ranking orders have been observed between η_o or σ_o as compared with the ranking of G'. That was to say, the SX-H paste was more stable and elastic under shear conditions. It was also showed that the η_o for the paste of SX-H was 1.4 times higher than the SX-U paste. The cross linkages between the starch and xanthan produced by dry heating could have been responsible for the high η_o and σ_o. While S-H paste had very low shear stability, which did not show a stable zero order Newtonian region. Dry heating might induce the disruption of molecular structure and hydrogen bonds between molecules inside the starch granules, resulting in the instability under shearing. With the addition of xanthan, the association between xanthan and starch made the paste more stable under heating and shearing.

From the study above, the pasting and rheological properties of dry heated starch–xanthan system were quite different from the simple starch–xanthan mixture. In order to elucidate more information of the interactions between the starch and the xanthan after dry heat treatment, several additional tests were carried out.

3.3. Particle size distributions

Particle size distributions of S-U, S-H, SX-U, and SX-H were determined (Fig. 3) to investigate the changes in granule size and possible aggregation effects from the dry heating. The SX-U dispersion displayed almost the same particle size distribution as the S-U. The average particle size of these two samples was around 5.95 μm. And the particle size distribution of S-H was also similar to those of S-U. Dramatic change in particle size was found for the SX-H sample. The shape of the particle size distribution changed from single to bi-modal peaked distribution, resulting in significant increase in average particle size. The average particle size of the first peak was around 6.96 μm, and that of the second peak was around 72.85 μm. The particle size distribution of the SX-H mixture suggested that several starch chains and granules were linked to a xanthan molecule, leading to several times increase of the relative particle size. If two or more starch chains were connected to a xanthan molecule, the xanthan molecule may have acted as a granular cross-linking agent. The degree of cross-linking of starch chains to a xanthan molecule would be an important determinant of pasting behavior (Lim et al., 2003). As could been observed from the pasting curves in Fig. 1, the SX-H showed the pasting characteristic of cross-linking. It suggested that dry heating of starch and xanthan appeared to connect xanthan polymers both intra- and inter- to the starch granules. The intra-granular cross-linking was thought to restrict the granular swelling during pasting, and the inter-granular cross-linking provided an increase in the observed particle size distributions.

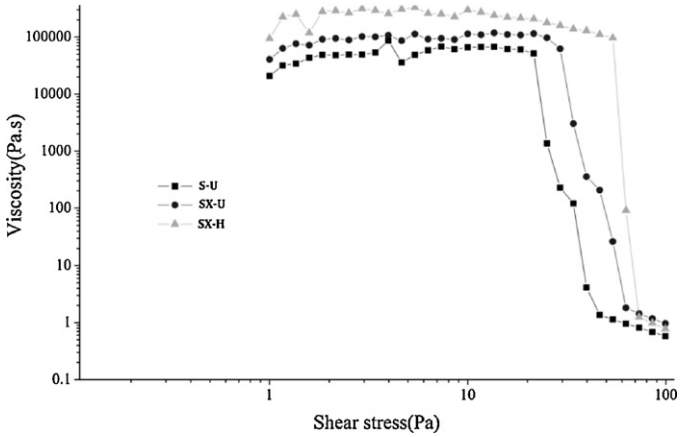

Fig. 2. Flow curves of waxy rice starch (S) and waxy rice starch–xanthan mixture (SX) either heated (-H) or unheated (-U) in a dry state.

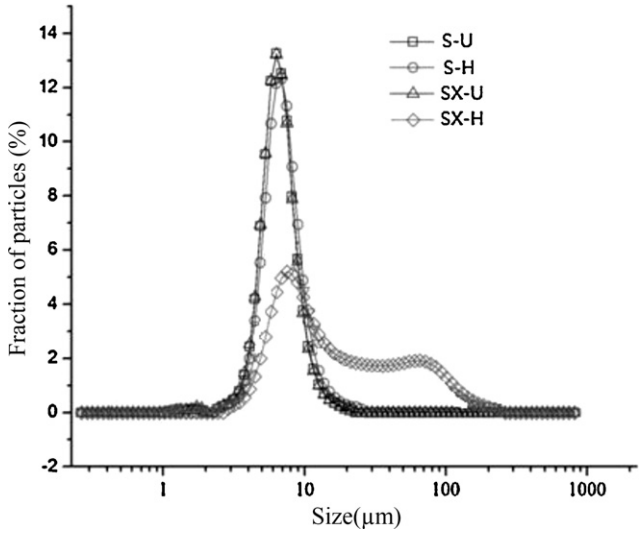

Fig. 3. Particle size distributions of waxy rice starch (S) and waxy rice starch–xanthan mixture (SX) either heated (-H) or unheated (-U) in a dry state.

Table 3
Degree of crystallinity and thermal properties of waxy rice starch (S) and waxy rice starch-xanthan mixture (SX) either heated (-H) or unheated (-U) in a dry state.

Samples	Degree of Crystallinity (%)	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)
S-U	46.18 ± 0.95^{a}	64.6 ± 0.5^{ab}	69.5 ± 0.7^{a}	75.3 ± 0.6^{a}	10.3 ± 0.4^{a}
S-H	55.29 ± 0.87^{c}	62.5 ± 0.2^{c}	67.4 ± 0.4^{b}	74.7 ± 0.4^{a}	11.2 ± 0.2^{b}
SX-U	48.11 ± 0.83^{b}	64.6 ± 0.3^{a}	69.5 ± 0.0^{a}	76.8 ± 0.1^{b}	8.1 ± 0.4^{d}
SX-H	48.94 ± 0.86^{b}	63.4 ± 0.6^{bc}	68.8 ± 0.5^{a}	76.7 ± 0.5^{b}	9.0 ± 0.3^{c}

Values are means ± SD of triplicate measurements.
Values in the same column with different superscripts are significantly different ($p \leq 0.05$).

3.4. X-ray diffraction

The X-ray diffraction patterns of all the samples were of the 'A' type (Phothiset & Charoenrein, 2007) which is representative of grain starches with main reflections at 2θ of 15°, 17°, 18° and 23.5°,respectively (Fig. 4). Although the crystallinity increased slightly after dry heating (Table 3), no other changes were apparent from the spectral patterns (Fig. 4). Kuakpetoon and Wang (2006) chemically modified corn starch with NaOCl, and found an increase of crystallinity which was due to the degradation of amorphous region. Here, the crystallinity of the starch granules increased after a dry heat treatment, suggesting a partial reorganization of the amorphous region during dry heating. Previous studies have shown that X-ray intensities of cereal starches (A type) increased with heat-moisture treatments (Hoover & Vasanthan, 1994). This study showed that under low moisture heat treatment, crystallinity can also increase. It suggested that the amorphous area was more sensitive to heat treatment than the crystalline region whether under low or high moisture content. But with the addition of xanthan, no significant difference of crystallinity was observed between the SX-U and SX-H mixtures (Table 3), illustrating that the amorphous region of the granule became more resistant to dry heating with the addition of xanthan.

3.5. Thermal properties

Gelatinization properties of S-U, S-H, SX-U, and SX-H were measured by DSC (Table 3). The gelatinization onset (T_o) and gelatinization temperatures (T_p) for SX-H were lower than that of the S-U, which were consistent with that of the pasting onset temperatures (Table 1). The melting enthalpy (ΔH) was higher for the S-H as compared to S-U. The increase of crystallinity in the starch granule would be account for the increase in ΔH. Thermal changes of amorphous region during dry heating resulted in higher crystallinity, which provided a higher melting enthalpy. It was noticed that ΔH of both SX-U and SX-H was lower than that of S-U and S-H (Table 3). The drop in ΔH may be due to the missing starch in SX-U and SX-H, since they were blends of starch and xanthan, and xanthan would not absorb heat to melt during starch gelatinization. No difference was found for T_p between the samples of S-U, SX-U, and SX-H. But the value of T_c was slightly higher for both SX-U and SX-H. The immobilization of water molecules by xanthan was the cause of the increase in T_c. Khanna and Tester (2006) also reported that T_o and T_p of normal starches were minimally affected by the addition of Konjac glucomannan (when the moisture content was relatively high), but T_c shifted to higher values with the increase of the Konjac glucomannan content. Dramatic reduction in ΔH was observed for SX-U. A similar result was reported by Chung et al. (2007)who found that adding xanthan to a starch–phosphate mixture prior to dry heat treatment resulted in reductions in the melting enthalpy. They suggested that it was related to the interactions between starch molecules and xanthan. There was no significant change in the gelatinization temperatures for the SX-H, but ΔH was increased. It indicated that the thermal stability of starch–xanthan blend was strengthened by dry heat treatment.

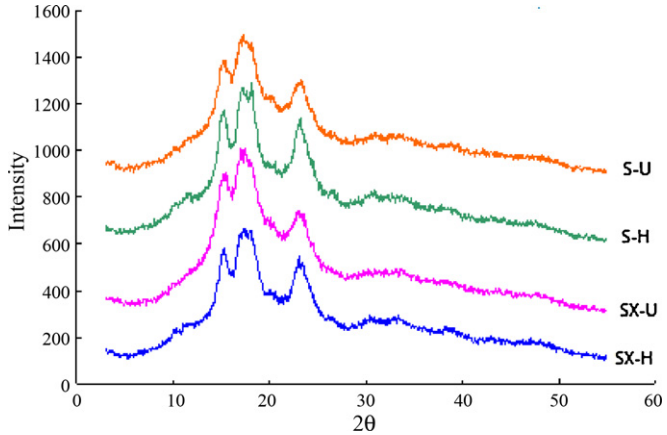

Fig. 4. X-ray diffraction patterns of waxy rice starch (S) and waxy rice starch–xanthan mixture (SX) either heated (-H) or unheated (-U) in a dry state.

4. Conclusions

The pasting and rheological properties of waxy rice starch were greatly changed by the heat treatment in a dry state with xanthan. The SX-H showed shear-stabilization during pasting, and the swelling of the granule was restricted. The paste of SX-H had a significantly higher zero order Newtonian viscosities and yield stress. Both G' and G'' increased, and tan δ was reduced for the SX-H. Thus, the gel forming ability of the waxy rice starch was strengthened after dry heat treatment with xanthan. An increase in starch particle size of SX-H suggested a cross linking of the starch granules by the xanthan polymers. The dry heat treatment appeared to crosslink xanthan polymers both intra- and inter-granular to the starch granules. An increase of crystallinity was observed for the starch after dry heat treatment, but with the addition of xanthan the amorphous region of the granule became more resistant to dry-heating. DSC analysis showed that the dry heat treatment raised the ΔH value of the rice starch, and the melting enthalpy was found to be correlated with the crystallinity. Xanthan could be an effective cross linking agent for the waxy rice starch.

Acknowledgments

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