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Effects of cellulose derivatives and carrageenans on the pasting, paste, and gel properties of rice starches

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Abstract

Effects of different cellulose derivatives and carrageenans on the pasting, rheological, and textural properties of normal (NRS) and waxy (WRS) rice starches were investigated. When suspensions of both NRS and WRS were heated in a Rapid Visco Analyser (RVA) in the presence of the hydrocolloids, increases in apparent pasting temperatures and peak and final viscosities in the following decreasing order were observed: methylcellulose (MC) > carboxymethylcellulose (CMC) for cellulose derivatives and λ- > ί- > κ-carrageenan for carrageenans. Slight decreases in peak and final viscosities were observed when hydroxypropylmethylcellulose (HPMC) was the hydrocolloid. Dynamic viscoelasticity measurements indicated that NRS–hydrocolloid pastes were less solid-like than the control, as evidenced by their higher tan δ values, whereas tan δ values of WRS–hydrocolloid pastes were the same as that of the control. Steady shear rheological measurements showed that addition of the different hydrocolloids used increased the apparent viscosity ($\eta_{a,100}$) and consistency coefficient (K) values of both starches with the same trend as that observed during pasting, whereas the opposite trend was observed for the flow behavior index (n) values. The hardness and adhesiveness of NRS pastes were significantly increased by addition of κ- and ί-carrageenans, but were unaffected by the other hydrocolloids. A similar effect was observed for WRS, with the exception of κ-carregeenan, in which the hardness of the mixed paste was decreased. The starch–hydrocolloid pastes exhibited a phase-separated microstructure in which amylose- and amylopectin-rich domains were dispersed in a hydrocolloid-rich continuous phase.

Keywords: Rice starch; Cellulose derivatives; Carrageenan; Pasting properties; Rheology; Texture

1. Introduction

Rice (*Oryza sativa* L.) starch is used in a wide range of food products, either as a raw material or as a food additive. Rice starch, as other starches, is composed of amylose and amylopectin. This starch can be classified into four groups according to its amylose content: low-amylose (12–20%), intermediate amylose (20–25%), high-amylose (>25%), and waxy (little to no amylose) rice starches. These starches have many unique attributes that make them some of the most interesting starches in the food industry (Bao & Bergman, 2004). However, rice starches, in common with other cereal starches, have negative aspects, such as gel syneresis, retrogradation, and tendency to exhibit breakdown, either from extended cooking, high shear, or acidic conditions, producing weak-bodied, cohesive, rubbery pastes, and undesirable gels (Whistler & BeMiller, 1997). This is usually undesirable and can be controlled by chemical modifications (Wurzburg, 1986). An alternative to the expensive chemical modification to overcome these shortcomings is the blending of native starches with polysaccharide hydrocolloids as reviewed by Appelqvist and Debet (1997). These mixtures are increasingly important ingredients in the modern health-conscious food industry. Cellulose derivatives and carrageenans are among the most commonly used as natural food gums in real food systems

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(Thomas, 1997; Whistler & BeMiller, 1997; Zecher & Gerrish, 1997).

Cellulose derivatives such as carboxymethylcellulose (CMC), methylcellulose (MC), and hydroxypropylmethylcellulose (HPMC) are water-soluble cellulose ethers (Zecher & Gerrish, 1997). These compounds are compatible with a wide range of other food ingredients, including starches, over a wide concentration range. In spite of this fact, there are only a few reports on the effect of these cellulose derivatives on pasting (Christianson, Hodge, Osborne, & Detroy, 1981; Lee, Baek, Cha, Park, & Lim, 2002; Naruenartwongsakul, Chinnan, Bhumiratana, & Yoovidhya, 2004; Rojas, Rosell, & Benedito de Barber, 1999; Shi & BeMiller, 2002) and rheological (Eidam, Kulicke, Kuhn, & Stute, 1995; Peressini, Bravin, Lapasin, Rizzotti, & Sensidoni, 2003) properties of starches. Moreover, to the best of our knowledge, there is no information available on the comparison of the effects of different cellulose derivatives on both pasting and rheological properties of normal and waxy rice starches.

Carrageenans (sulfated polysaccharides obtained from red seaweeds) are frequently used in combination with starch in various food products (Thomas, 1997). However, again there are only a few reports on the effect of carrageenans on pasting (Lee et al., 2002; Rojas et al., 1999; Shi & BeMiller, 2002), rheological (Autio, Vesterinen, & Stolt, 2002; Eidam et al., 1995; Lafargue, Lourdin, & Doublier, 2007; Savary, Handschin, Conde-Petit, Cayot, & Doublier, in press; Tecante & Doublier, 1999; Tischer, Noseda, Freitas, Sierakowski, & Duarte, 2006; Tye, 1988), and textural (Huang, Kennedy, Li, Xu, & Xie, 2007) properties of different starches. When compared to other food hydrocolloids such as guar gum and xanthan gum, the number of studies on carrageenans in combination with starches is still limited and most have focused on κ-carrageenan. In addition, the effect of different carrageenans on both pasting and rheological properties of normal and waxy rice starches has not been investigated.

The objective of this study was to investigate the effects of different types of cellulose derivatives (CMC, MC, and HPMC) and carrageenans (κ-, ί-, and λ-carrageenans) on the pasting and paste (rheological and textural) properties of normal and waxy rice starches by Rapid Visco Analysis (RVA), swelling and solubility measurements, dynamic and steady shear measurements, texture profile analysis, and light microscopy.

2. Materials and methods

2.1. Materials

Normal and waxy rice starches were obtained from California Natural Products (Lathrop, CA, USA) and A&B Ingredients (Fairfield, NJ, USA), respectively. As the normal rice starch contained only 11.9% amylose, it is a low-amylose rice starch. Moisture (AACC, 2000, Method 44–19) and amylose (AACC, 2000, Method 61–03) contents of the normal and waxy rice starches were 10.6 and 11.9 wt%, and 10.7 and 0.92 wt%, respectively. Carboxymethylcellulose (CMC) (Hercules Inc., DE, USA), methylcellulose (MC) (Dow Chemical Co., MI, USA), hydroxypropylmethylcellulose (HPMC) (Dow Chemical Co., MI, USA), κ-carrageenan (Sigma–Aldrich Co., MO, USA), ί-carrageenan (Sigma–Aldrich Co., MO, USA), and λ-carrageenan (FMC Corporation, PA, USA) were commercial samples.

2.2. Determination of pasting and paste properties

Pasting and paste properties of the normal and waxy rice starches were determined in the presence and absence of the hydrocolloids by a Rapid Visco Analyser (Model RVA-4C, Newport Scientific Pty. Ltd., Warriewood, Australia). The starch alone slurry (8 wt%) was prepared by dispersing weighed amounts of the starch (dry basis) in distilled water. When hydrocolloids were used, the starch (7.2 wt%) and the hydrocolloid powder (0.8 %) were thoroughly dry blended prior to slurrying in water. In both cases, the slurries were stirred for 1 h at room temperature to avoid clump formation. The slurries (25 g) were then poured into aluminum canisters and stirred manually using plastic paddles for 20–30 s before insertion into the RVA machine. The heating and cooling cycles were programmed following the general pasting method (STD 1). The slurry was held at 50 °C for 1 min, heated to 95 °C within 3 min 42 s and then held at 95 °C for 2 min 30 s. It was subsequently cooled to 50 °C within 3 min 48 s and held at 50 °C for 2 min, while maintaining a rotation speed of 160 rpm. Thermocline for Windows (version 2.2, 1999) was used to give the viscosity in mPa s units.

2.3. Determination of swelling power and solubility index

The concentration of starch used in this experiment was less than the close packing concentration (~2.0%) of starch granules (Vandeputte, Derycke, Geeroms, & Delcour, 2003). The starch alone (1.25 wt%, db) or starch–hydrocolloid (1.125 wt% starch and 0.125 wt% hydrocolloid, both dry basis) suspensions were put into 50-mL centrifuge tubes and heated in a boiling water bath for 10 min with minimum shear conditions. After heating, the centrifuge tubes containing the samples were cooled in an ice bath for 5 min and then centrifuged at 7000*g* at 25 °C for 15 min. The supernatant was removed for the measurement of dissolved starch by drying to constant weight in a hot air oven at 105 °C. Precipitated paste and dried supernatant were weighed. The swelling power (SP) and solubility index (SOL) were calculated based on the assumption that the total amount of gum remained in the supernatant. The SP is the ratio of the wet weight of precipitated starch gel to its dry weight. The SOL is the percentage of dried mass of solubles in the supernatant (after subtraction of the dry

weight of gum added) to the dry mass of the whole starch sample.

2.4. Rheological measurements

Dynamic viscoelastic and steady flow properties of the freshly prepared pastes of starch alone and starch–hydrocolloid mixtures obtained from pasting in the RVA which were determined after holding at room temperature (~25 °C) for 1 h by using a rheometer (Viscotech DSR, Rheologica Instruments Inc., NJ, USA) with a cone and plate geometry sensor (2° cone angle, 50 mm diameter, and 0.05 mm gap). The sample was placed into the rheometer which was equilibrated to 30 °C. Two dynamic viscoelastic measurements were made: (1) deformation sweeps at a constant frequency (10 Hz) to determine the maximum deformation attainable by a sample in the linear viscoelastic range, and (2) frequency sweeps over a range of 1–100 Hz at a constant deformation (0.5% strain) within the linear viscoelastic range. The storage modulus (G'), loss modulus G'' complex viscosity ($\eta^* = \sqrt{(G')^2 + (G'')^2}/\omega$), and loss tangent ($\tan\delta = G''/G'$) as a function of frequency (ω) were obtained.

Steady flow tests were also performed on the freshly prepared paste samples at 30 °C to obtain shear rate versus shear stress data. The cone was programmed to increase the shear rate from 1 to 500 s^{-1} (up curve) in 400 s followed immediately by a reduction from 500 to 1 s^{-1} in 400 s (down curve). Data from the down curve of the shear cycle were used to characterize the flow of the paste samples and to estimate the power law parameters by using the equation $\sigma = K\dot{\gamma}^n$, where σ is the shear stress (Pa), $\dot{\gamma}$ is the shear rate (s^{-1}), K is the consistency coefficient (Pa s^n), and n is the dimensionless flow behavior index.

2.5. Texture profile analysis

Texture profile analyses of the freshly prepared paste samples (8 wt%) were performed at room temperature (~25 °C) using a TA.XT2i Texture Analyzer (Stable Micro Systems Ltd., Surrey, UK) equipped with Texture Expert for Windows version 1 software; a 2 kg load was used for force calibration. Weighed paste samples (~25 g) were poured into cylindrical containers (35 mm internal diameter, 65 mm height) and kept at room temperature (~25 °C) for 1 h prior to measurement. One compression cycle was applied using a hemispherical probe (P/0.5HS) at a constant crosshead velocity of 1 mm s^{-1} to a sample depth of 15 mm (50% gel height), followed by return to the original position. From the resulting force-time curve, the values for texture attributes, i.e., hardness and adhesiveness, of the paste samples were measured according to the definitions of Pons and Fiszman (1996). Hardness is defined as the peak force observed during the compression cycle. Adhesiveness is the negative force area representing the work necessary to pull the compressing plunger away from the sample.

2.6. Microscopic examination

Freshly prepared paste samples (8 wt%) obtained from the RVA analysis were cooled down and kept at room temperature for 1 h prior to examination. The samples were placed on microscope slides, stained with Lugol's solution (333 mg I_2 + 667 mg KI in 100 mL water), and then covered with cover glasses. The microstructure of the paste samples was examined using a Leitz Laborlux 12 Pol microscope (Ernst Leitz Wetzlar GMBH, Wetzlar, Germany) at 100× magnification.

2.7. Statistical analysis

All measurements were made in triplicate for each sample. Results are expressed as means ± standard deviations. A one-way analysis of variance (ANOVA) and Tukey's test were used to establish the significance of differences among the mean values at 0.05 level of confidence. The statistical analyses were performed using SPSS version 12.0 for Windows program (SPSS Inc., Chicago, IL, USA).

3. Results and discussion

3.1. Pasting properties

The pasting and paste characteristics of the normal rice starch (NRS) and waxy rice starch (WRS) in the absence and presence of different cellulose derivatives and carrageenans determined by RVA analysis are summarized in Tables 1 and 2, respectively. The NRS alone exhibited higher peak, breakdown, final, and setback viscosities and lower pasting temperature than the WRS alone. This could be attributed to the fact that the WRS (with <1% apparent amylose) had a much higher swelling power than the NRS (see Section 3.2), leading to a lower rigidity of the starch granular structure (Lii, Tsai, & Tseng, 1996). In a concentrated regime, i.e., one which is higher than an average close packing concentration (2.0% [75–125 °C] and 3.0% [95–125 °C] for the WRS and NRS, respectively), the viscosities of starch suspensions during pasting are mainly determined by the particle rigidity of the swollen granules (Vandeputte et al., 2003). It can be concluded that the pasting properties of starch depended mainly on interactions among the close-packed granules and their rigidity during the heating process.

Addition of cellulose derivatives and carrageenans to both NRS and WRS generally resulted in a significant ($P \leqslant 0.05$) increase in peak, breakdown, final, and setback viscosities and pasting temperatures, with the exception of HPMC, which did not significantly affect the pasting properties of these starches. NRS and WRS were affected to different degrees by the different types of hydrocolloids. The

Table 1
Pasting properties of normal rice starch (7.2 wt%) in the presence of various hydrocolloids (0.8 wt%)[1]

Hydrocolloid	Peak viscosity (mPa s)	Breakdown (mPa s)	Final viscosity (mPa s)	Setback (mPa s)	Pasting temperature (°C)
None (control)[2]	2430 ± 28^{e}	944 ± 19^{de}	1693 ± 19^{c}	207 ± 10^{bc}	67.0 ± 0.2^{ef}
CMC	3382 ± 136^{c}	1313 ± 76^{c}	2517 ± 16^{a}	448 ± 60^{a}	70.2 ± 0.1^{cd}
MC	4351 ± 185^{a}	2378 ± 305^{a}	2221 ± 223^{b}	248 ± 23^{b}	66.5 ± 0.5^{f}
HPMC	2406 ± 90^{e}	1048 ± 60^{cd}	1488 ± 16^{c}	130 ± 35^{c}	68.9 ± 0.5^{de}
κ-Carrageenan	2615 ± 13^{e}	602 ± 39^{e}	2187 ± 21^{b}	174 ± 9^{bc}	71.6 ± 0.5^{bc}
í-Carrageenan	3096 ± 89^{d}	1132 ± 31^{cd}	2377 ± 37^{ab}	440 ± 21^{a}	73.5 ± 0.8^{ab}
λ-Carrageenan	3991 ± 23^{b}	1948 ± 60^{b}	2509 ± 41^{a}	465 ± 17^{a}	74.2 ± 1.6^{a}

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] 8 wt% normal rice starch (NRS).

Table 2
Pasting properties of waxy rice starch (7.2 wt%) in the presence of various hydrocolloids (0.8 wt%)[1]

Hydrocolloid	Peak viscosity (mPa s)	Breakdown (mPa s)	Final viscosity (mPa s)	Setback (mPa s)	Pasting temperature (°C)
None (control)[2]	1414 ± 17^{e}	660 ± 11^{d}	895 ± 9^{e}	141 ± 4^{c}	69.1 ± 0.4^{d}
CMC	2327 ± 41^{c}	1142 ± 27^{d}	1632 ± 14^{b}	447 ± 6^{a}	71.6 ± 0.5^{c}
MC	3122 ± 128^{a}	1812 ± 143^{a}	1550 ± 60^{c}	239 ± 31^{b}	72.4 ± 0.5^{bc}
HPMC	1513 ± 16^{e}	772 ± 25^{cd}	871 ± 18^{e}	130 ± 11^{c}	70.6 ± 1.0^{cd}
κ-Carrageenan	1884 ± 27^{d}	806 ± 16^{cd}	1332 ± 15^{d}	154 ± 9^{b}	71.8 ± 0.9^{c}
í-Carrageenan	2197 ± 29^{c}	925 ± 30^{c}	1686 ± 17^{b}	414 ± 9^{a}	74.3 ± 0.8^{ab}
λ-Carrageenan	2759 ± 17^{b}	1254 ± 40^{b}	1932 ± 12^{a}	428 ± 20^{a}	75.4 ± 1.0^{a}

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] 8 wt% waxy rice starch (WRS).

trend of this effect was MC > CMC > HPMC for cellulose derivatives and λ-carrageenan (λC) > í-carrageenan (íC) > κ-carrageenan (κC) for carrageenans. This result can be interpreted by assuming that the system is biphasic, with the hydrocolloid located entirely in the continuous phase. Its concentration would then increase as the volume of the phase accessible to the hydrocolloid was reduced due to swelling of the starch granules during pasting. This resulted in a pronounced increase in the viscosity of the continuous phase and in turn the overall viscosity of the suspension itself owing to the thickening properties of these hydrocolloids (Alloncle, Lefebvre, Llamas, & Doublier, 1989) added to the thickening produced by swollen starch granules. Therefore, the thickening capacity of each tested hydrocolloid could dominate the viscosity of the starch–hydrocolloid pastes. This hypothesis was confirmed by the fact that the viscosity of each hydrocolloid solution decreases in the following order: MC > CMC > HPMC (Naruenartwongsakul et al., 2004) and λC > íC > κC at a temperature higher than 40–60 °C (Imeson, 2000). It has also been proposed that the increase in viscosity of starch–hydrocolloid systems is due to interactions between leached amylose and low-molecular weight amylopectin molecules and hydrocolloids (Bahnassey & Breene, 1994; Christianson et al., 1981; Shi & BeMiller, 2002), and that these are a function of the structure of hydrocolloid, so structural and rheological properties of the hydrocolloid are very relevant. Possibly, both of these mechanisms are involved.

3.2. Swelling power and solubility index

The effect of hydrocolloid addition on swelling power (SP) and solubility index (SOL) of NRS and WRS is shown in Table 3. SP and SOL of WRS alone were, as expected higher than those of NRS, as the swelling behavior of cereal starch is primarily the property of its amylopectin content and amylose acts as both a diluent and an inhibitor of swelling (Tester & Morrison, 1990a). Maximal swelling might also be related to the molecular weights and the shapes of the amylopectin molecules (Tester & Morrison, 1990b). Lii et al. (1996) observed that WRS granules were less firm and tended to disintegrate easily while swollen and extensively overcrowded. In contrast, NRS granules were more rigid, less swelling, and not easily ruptured. Generally, the amount of exudate from NRS granules was small, suggesting that the main component leached from the granules was amylose (Mandala & Bayas, 2004), whereas, the amount of exudate from WRS granules was large and consisted of amylopectin molecules (Tester & Morrison, 1990a).

Addition of the tested hydrocolloids to NRS slightly increased SP, but significantly ($P \leqslant 0.05$) promoted the leaching of amylose at temperatures above the gelatinization temperature. According to Christianson et al. (1981), the addition of hydrocolloids can make the shear forces exerted on the swollen granules in the shear field much larger than those encountered in the starch–water suspensions. This can enhance water uptake (increasing

Table 3
Swelling power and solubility index of normal and waxy rice starches (1.125 wt%) in the presence of various hydrocolloids (0.125 wt%) heated at 100 °C for 10 min under low-shear conditions[1]

Hydrocolloid	Normal rice starch		Waxy rice starch	
	Swelling power (g/g)[2]	Solubility index (%)	Swelling power (g/g)[2]	Solubility index (%)
None (control)[3]	15.5 ± 0.7^{b}	3.9 ± 0.6^{d}	26.9 ± 2.6^{b}	14.9 ± 2.8^{a}
CMC	22.7 ± 0.2^{a}	13.8 ± 0.1^{a}	31.0 ± 1.6^{a}	12.9 ± 2.2^{ab}
MC	17.4 ± 0.7^{ab}	12.6 ± 0.9^{abc}	19.1 ± 0.4^{c}	9.1 ± 0.5^{bc}
HPMC	17.3 ± 2.8^{ab}	12.3 ± 0.4^{c}	26.3 ± 0.7^{b}	11.2 ± 0.5^{abc}
κ-Carrageenan	20.6 ± 1.2^{ab}	13.7 ± 0.2^{ab}	28.2 ± 0.7^{ab}	13.1 ± 0.8^{ab}
ί-Carrageenan	18.7 ± 1.7^{ab}	12.4 ± 0.0^{bc}	31.7 ± 1.3^{a}	10.0 ± 1.9^{bc}
λ-Carrageenan	21.6 ± 5.1^{ab}	12.6 ± 0.6^{abc}	26.0 ± 0.1^{b}	8.3 ± 0.5^{c}

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] Grams of starch gel per gram of its dry weight.
[3] 1.25 wt% normal rice starch (NRS) or waxy rice starch (WRS).

swelling), the breakdown of granules and the amount of material exuded into the continuous phase. This effect was not much different among the different hydrocolloids, indicating that SP and SOL of NRS were affected to the same degree by the different tested hydrocolloids. It was previously reported that SP of starch granules during heating was enhanced by the presence of hydrocolloids (Chaisawang & Suphantharika, 2006; Mandala & Bayas, 2004) and that SOL was enhanced up to a temperature of 80 °C (Mandala & Bayas, 2004).

Addition of hydrocolloids to WRS produced a variety of effects on its SP during heating, i.e., with different hydrocolloids, it increased, decreased, or had no effect, whereas SOL tended to decrease. This evidence could be explained by the fact that the swollen WRS granules were less firm and might be more disintegrated, even in the low-shear environment of the experiment, and therefore, addition of hydrocolloids did not increase SOL of the starch–hydrocolloid systems. The different effect of each tested hydrocolloid on SP of WRS demonstrated different interactions of each hydrocolloid and WRS, which in turn depended upon the chemical structure of the added hydrocolloids. The presence of hydrocolloid was found to increase the granule size (swelling) of waxy corn starch and crosslinked waxy corn starch during heating (Achayuthakan, Suphantharika, & Rao, 2006; Tecante & Doublier, 1999).

3.3. Dynamic rheological properties

The dynamic rheological properties of NRS and WRS in the absence and presence of different hydrocolloids are shown in Tables 4 and 5, respectively. In general, all pastes displayed a typical gel behavior (Clark & Ross-Murphy, 1987) where G' is higher than G'' throughout the measured frequency range and G' is almost independent of frequency (data not shown). The G' value of the NRS alone paste was 1.7 times greater than that of the WRS alone paste. The G' values of the NRS pastes prepared with added hydrocolloids were 1.1–1.6 times greater than the G' values of the WRS pastes prepared with added hydrocolloids, with the exception of κ-carrageenan which increased the G' value of the NRS paste to 12 times that of the NRS alone paste and 8.8 times that of the WRS–κ-carrageenan paste, indicating a much greater elasticity (gel-like character) of the NRS–κ-carrageenan paste via a κ-carrageenan–amylose interaction.

In the case of tan δ, the value for the WRS alone paste was 4.75 times that for the NRS alone paste. When hydrocolloids were present, the values for the WRS–hydrocolloid pastes were only slightly greater (1.1–1.25) than those for the NRS–hydrocolloid pastes, with the exceptions of the pastes containing MC and HPMC, in which cases the tan δ values of NRS–hydrocolloid pastes were 1.3 and 2.6

Table 4
Dynamic rheological properties of normal rice starch (7.2 wt%) and various hydrocolloids (0.8 wt%) gels measured at 10 Hz, 0.5% strain, and 30 °C[1,2]

Hydrocolloid	G' (Pa)	G' (Pa)	η^* (Pa s)	tan δ
None (control)[3]	128.0 ± 11.4^{b}	4.8 ± 2.1^{b}	2.1 ± 0.3^{b}	0.04 ± 0.01^{d}
CMC	154.0 ± 18.0^{b}	22.0 ± 7.4^{b}	2.5 ± 0.3^{b}	0.14 ± 0.03^{bcd}
MC	192.7 ± 17.6^{b}	49.9 ± 3.4^{b}	3.2 ± 0.3^{b}	0.26 ± 0.02^{ab}
HPMC	99.7 ± 31.1^{b}	28.9 ± 4.1^{b}	1.6 ± 0.5^{b}	0.31 ± 0.12^{a}
κ-Carrageenan	1513.3 ± 350.2^{a}	241.3 ± 62.1^{a}	24.4 ± 5.7^{a}	0.16 ± 0.01^{bcd}
ί-Carrageenan	310.0 ± 37.7^{b}	34.5 ± 2.4^{b}	5.0 ± 0.6^{b}	0.11 ± 0.01^{cd}
λ-Carrageenan	186.3 ± 8.1^{b}	37.0 ± 2.0^{b}	3.0 ± 0.1^{b}	0.20 ± 0.02^{abc}

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] G', storage modulus; G'', loss modulus; η^*, complex viscosity; tan δ, loss tangent.
[3] 8 wt% normal rice starch (NRS).

Table 5
Dynamic rheological properties of waxy rice starch (7.2 wt%) and various hydrocolloids (0.8 wt%) gels measured at 10 Hz, 0.5% strain, and 30 °C[1,2]

Hydrocolloid	G' (Pa)	G'' (Pa)	η^* (Pa s)	tan δ
None (control)[3]	74.6 ± 15.2^{d}	13.9 ± 1.4^{de}	1.2 ± 0.2^{d}	0.19 ± 0.03^{ab}
CMC	109.0 ± 5.0^{cd}	18.6 ± 3.4^{cd}	1.8 ± 0.1^{cd}	0.17 ± 0.04^{abc}
MC	123.0 ± 20.1^{c}	24.4 ± 7.7^{bc}	2.0 ± 0.3^{c}	0.20 ± 0.03^{a}
HPMC	66.9 ± 17.4^{d}	8.1 ± 2.3^{e}	1.1 ± 0.3^{d}	0.12 ± 0.03^{c}
κ-Carrageenan	172.0 ± 25.5^{b}	35.1 ± 3.3^{a}	2.8 ± 0.4^{b}	0.20 ± 0.01^{a}
ί-Carrageenan	271.0 ± 4.4^{a}	33.3 ± 0.7^{ab}	4.4 ± 0.1^{a}	0.12 ± 0.01^{bc}
λ-Carrageenan	145.7 ± 11.9^{bc}	34.0 ± 0.5^{ab}	2.4 ± 0.2^{bc}	0.23 ± 0.02^{a}

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] G', storage modulus; G'', loss modulus; η^*, complex viscosity; tan δ, loss tangent.
[3] 8 wt% waxy rice starch (WRS).

times greater than those for the WRS–hydrocolloids pastes, respectively. These results can be interpreted as follows. The weaker and more highly swollen WRS granules (Table 3) produce a less rigid paste, as compared to NRS granules. When CMC and the carrageenans are added to NRS, the tan δ values are increased 2.75–5.0 times, i.e., reducing the rigidity of the pastes, making them more like the WRS pastes, perhaps indicating a prevention of amylose retrogradation. The greatest increase (6.5–7.75 times) in tan δ values, over the control (NRS alone) paste, occurred with the NRS–MC and NRS–HPMC pastes, indicating that they had the greatest effect on decreasing paste rigidity, perhaps by preventing amylose retrogradation to the greatest degree; although since their tan δ values were even greater than those for WRS alone, there may be an additional factor. In the case of NRS and cellulose derivatives, reduced setback and final viscosities was found only for NRS–HPMC pastes (Table 2); however, it is difficult to interpret these results since MC and HPMC solutions can gel when heated and return to a fluid state when cooled and a weak gel (due to the low-concentration) can be disrupted by shear. Even so, HPMC was unique in that it was the only hydrocolloid that reduced final and setback viscosities over that of the control and did so with both starches (Tables 1 and 2).

Addition of all the tested hydrocolloids, with the exception of κC, did not significantly affect G', G'', and η^* values of NRS, whereas tan δ values increased (Table 4). The κC significantly ($P \leqslant 0.05$) increased G', G'', and η^* values of NRS pastes. According to the differences observed in tan δ values, the NRS alone paste seemed to be more structured and more solid-like in comparison with the NRS–hydrocolloid pastes. Additionally, amylose did not seem to interact with hydrocolloids in the continuous phase, and different polymers excluded each other. This finding supports the hypothesis that these systems involve mixtures in which intermolecular interactions between like molecules are favored, which locally at least results in mutual exclusion (Annable, Fitton, Harris, Phillips, & Williams, 1994). This result was confirmed by microscopic examination of the paste structure (Fig. 1) (to be discussed later). κ-Carrageenan was an exception. In contrast to all the other polysaccharides investigated, G' of the NRS–κC paste was more than 10 times higher than that of the NRS alone paste. It seems likely, therefore, that different interactions are involved that are specific to κC and rice amylose molecules. By combining rheological and turbidity measurements, Tecante and Doublier (2002) investigated the interaction and viscoelastic properties of the amylose–κC mixtures. It was found that, at a gel forming concentration of κC, i.e., between 0.72 and 0.92 wt%, at 25 °C, the κC formed the continuous phase in which amylose gel was dispersed and G' increased markedly showing a predominance of the properties of a κC gel. Eidam et al. (1995) reported that κC (at a concentration of 0.35%) had a gel-accelerating, network-weakening role on the gelation of completely gelatinized normal maize starch (6.65%), while ίC had a gel-retarding, network-strengthening effect. These systems were proposed to have exclusion and coupled network structures, originating from molecular incompatibility and specific interactions, respectively. Lai, Huang, and Lii (1999) reported that the gelation of both κC and ίC at lower concentrations (⩽2.0%) was accelerated by adding incompletely gelatinized rice starch, possibly due to some coupling between κC or ίC and soluble starch molecules. Microscopic examination of high-amylose maize starch–κC gels (3% total polysaccharide concentration) showed a phase-separated type of microstructure (Autio et al., 2002). The micrographs of mixed gels showed that when the ratio of κC to starch increased (up to 1% κC and 2% starch), the size of amylose-rich domains in the dispersed phase also increased. The discrepancy among these data could be attributed to several factors, such as the type and concentration of starch and hydrocolloid, amylose/amylopectin ratio of the starch, degree of gelatinization of the starch, and preparation of pastes or gels.

Addition of all the tested carrageenans and MC to WRS exhibited a significant ($P \leqslant 0.05$) increase in G', G'', and η^* values, whereas that of CMC and HPMC did not produce any significant effect on the viscoelastic behavior (Table 5). However, tan δ values of all the WRS–hydrocolloid pastes, except for the WRS–HPMC pastes, did not differ significantly from that of the control, indicating a similar microstructure of these pastes (Fig. 2). Due to an absence of

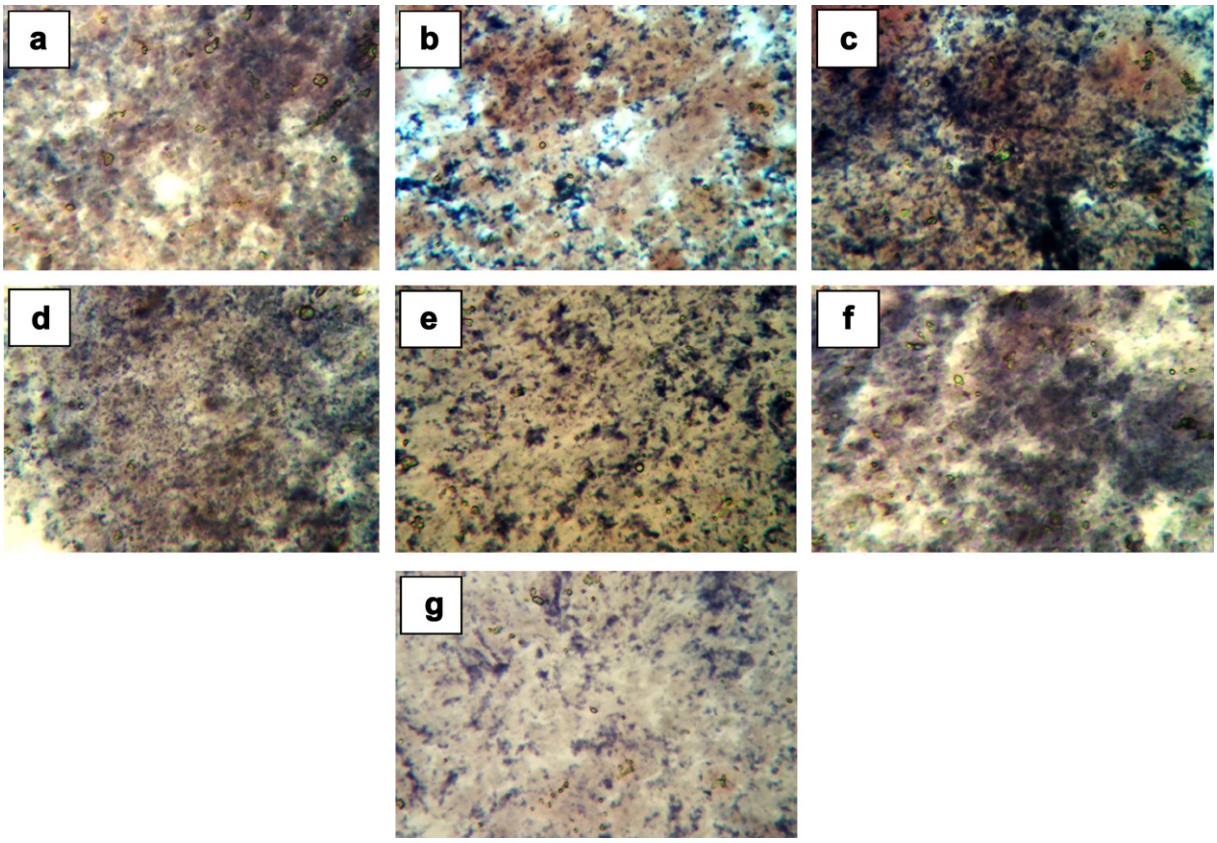

Fig. 1. Photographs of normal rice starch (NRS) (7.2 wt%) pastes produced in the presence of different hydrocolloids (0.8 wt%): (a) CMC, (b) MC, (c) HPMC, (d) κ-carrageenan, (e) ί-carrageenan, (f) λ-carrageenan, and (g) none (control). All pastes were stained with iodine and observed at 100× magnification.

amylose in the WRS paste, short-term retrogradation was absent and therefore, the addition of hydrocolloids could not alter the viscoelastic characteristics of the WRS pastes.

3.4. Steady shear rheological properties

For the range of shear rates used in this study, the power law model accurately described the flow behavior of each paste. Consistency coefficients (K) and flow behavior indices (n) along with coefficients of determination (R^2) and apparent viscosities at a shear rate of 100 s^{-1} ($\mu_{a,100}$) for the flow curves of NRS and WRS pastes in the absence or presence of the tested hydrocolloids are presented in Tables 6 and 7, respectively. All the pastes exhibited pseudoplastic, shear-thinning behavior, for which $n < 1$.

The NRS and WRS pastes exhibited similar steady shear rheological properties which in turn were affected by addition of different tested hydrocolloids in a similar way. The presence of hydrocolloids increased $\eta_{a,100}$ and K values of both NRS and WRS pastes in the following decreasing order: CMC ≈ MC > HPMC for cellulose derivatives, and λC ≈ ίC > κC for carrageenans, whereas the opposite trend was observed for n values. This result is in good agreement with the peak and final viscosities of the corresponding mixtures during pasting (Tables 1 and 2), indicating a predominant effect of the rheological properties of the added hydrocolloid as discussed earlier. In general, addition of carrageenans enhanced pseudoplasticity to a greater degree than did addition of cellulose derivatives as evidenced by their higher K values and lower n values.

3.5. Textural properties

The hardness and adhesiveness of NRS and WRS pastes with and without hydrocolloids are presented in Table 8. Addition of all cellulose derivatives at a concentration used in this experiment seemed to have no effect on the textural properties of both NRS and WRS pastes. On the contrary, addition of κC or ίC to NRS significantly ($P \leqslant 0.05$) increased the hardness and adhesiveness of the mixed pastes as compared with the NRS alone paste. The main reason might be that κC and ίC are both gelling polysaccharides which can form network structures and enhance the textural properties of the pastes. Therefore, it could be concluded that the hardness and adhesiveness of the NRS–κC and NRS–ίC pastes mainly came from the network structure of κC and ίC, respectively, rather than from retrogradation of the amylose. This result is in good agreement with that previously reported for the NRS–κC systems (Huang et al., 2007). However, there was no

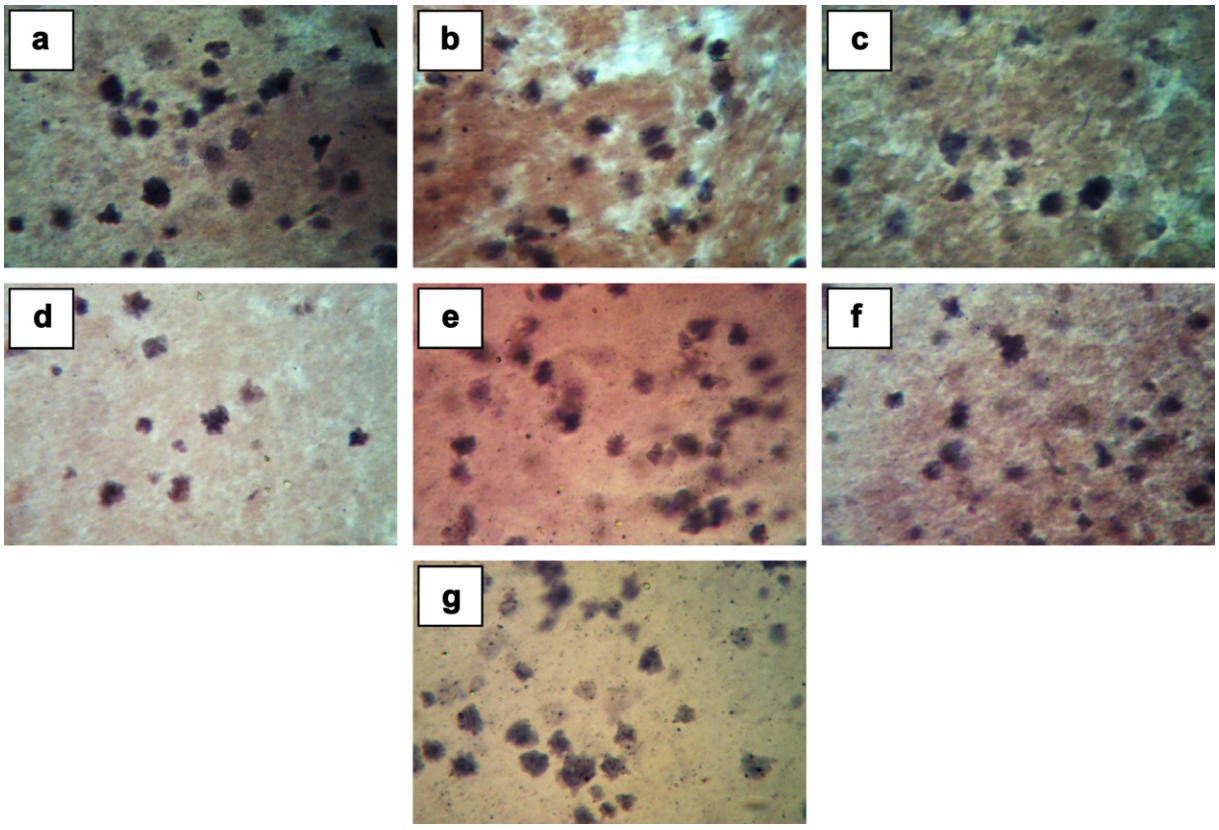

Fig. 2. Photographs of waxy rice starch (WRS) (7.2 wt%) pastes produced in the presence of different hydrocolloids (0.8 wt%): (a) CMC, (b) MC, (c) HPMC, (d) κ-carrageenan, (e) ί-carrageenan, (f) λ-carrageenan, and (g) none (control). All pastes were stained with iodine and observed at 100× magnification.

Table 6
Steady shear rheological properties of normal rice starch (7.2 wt%) and various hydrocolloids (0.8 wt%) gels measured at 30 °C[1,2]

Hydrocolloid	$\eta_{a,100}$ (Pa s)	K (Pa s^n)	n (−)	R^2
None (control)[3]	1.3 ± 0.0^{cd}	14.2 ± 0.0^{cd}	0.48 ± 0.01^{b}	0.9995
CMC	2.4 ± 0.3^{ab}	33.7 ± 6.6^{bc}	0.43 ± 0.01^{c}	0.9987
MC	2.1 ± 0.1^{abc}	22.4 ± 1.2^{cd}	0.49 ± 0.01^{ab}	0.9996
HPMC	1.0 ± 0.0^{d}	9.9 ± 0.2^{d}	0.51 ± 0.01^{a}	0.9996
κ-Carrageenan	1.5 ± 0.8^{bcd}	32.0 ± 19.8^{bcd}	0.36 ± 0.02^{d}	0.9889
ί-Carrageenan	2.6 ± 0.1^{a}	49.4 ± 3.7^{ab}	0.34 ± 0.01^{de}	0.9948
λ-Carrageenan	2.2 ± 0.1^{abc}	60.5 ± 4.0^{a}	0.33 ± 0.01^{e}	0.9989

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] $\eta_{a,100}$, apparent viscosity at $\dot{\gamma} = 100\ s^{-1}$; K, consistency coefficient; n, flow behavior index; R^2, coefficient of determination.
[3] 8 wt% normal rice starch (NRS).

Table 7
Steady shear rheological properties of waxy rice starch (7.2 wt%) and various hydrocolloids (0.8 wt%) gels measured at 30 °C[1,2]

Hydrocolloid	$\eta_{a,100}$ (Pa s)	K (Pa s^n)	n (−)	R^2
None (control)[3]	1.1 ± 0.0^{c}	14.3 ± 0.6^{d}	0.45 ± 0.01^{b}	0.9999
CMC	2.4 ± 0.1^{a}	32.9 ± 1.4^{b}	0.43 ± 0.01^{bc}	0.9991
MC	2.1 ± 0.1^{b}	23.1 ± 1.6^{c}	0.48 ± 0.01^{a}	0.9997
HPMC	0.9 ± 0.0^{c}	9.6 ± 0.3^{e}	0.49 ± 0.01^{a}	0.9996
κ-Carrageenan	2.1 ± 0.0^{b}	31.5 ± 0.7^{b}	0.42 ± 0.01^{c}	0.9989
ί-Carrageenan	2.4 ± 0.0^{a}	50.6 ± 2.7^{a}	0.35 ± 0.01^{d}	0.9927
λ-Carrageenan	2.5 ± 0.1^{a}	50.6 ± 1.3^{a}	0.35 ± 0.01^{d}	0.9988

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] $\eta_{a,100}$, apparent viscosity at $\dot{\gamma} = 100\ s^{-1}$; K, consistency coefficient; n, flow behavior index; R^2, coefficient of determination.
[3] 8 wt% waxy rice starch (WRS).

significant ($P \leqslant 0.05$) effect for the addition of λC to NRS possibly due to a non-gelling property of λC.

Surprisingly, addition of κC to WRS resulted in a significant decrease in the hardness and no significant effect on the adhesiveness of the paste, whereas the other carrageenans exhibited a similar effect as that observed in the case of NRS. This could be due to a specific interaction of amylose and κC which enhanced the gelling properties of κC as pointed out by Tecante and Doublier (2002). With amylose being essentially absent from WRS, κC could not form a gel network structure and had no effect on the texture of the mixed paste. In contrast, it is known that ίC has a synergistic interaction with all starches (Imeson, 2000; Thomas, 1997; Tischer et al., 2006), and it enhanced the textural properties of both NRS and WRS pastes. λ-Carrageenan did not affect the textural properties of the WRS–λC paste due to the fact that it is non-gelling.

Table 8
Textural properties of normal or waxy rice starches (7.2 wt%) and various hydrocolloids (0.8 wt%) gels measured at room temperature (25 °C)[1]

Hydrocolloid	Normal rice starch		Waxy rice starch	
	Hardness (g)	Adhesiveness (g mm)	Hardness (g)	Adhesiveness (g mm)
None (control)[2]	12.1 ± 0.4^{b}	16.2 ± 1.1^{c}	9.1 ± 0.3^{bc}	7.5 ± 2.0^{cd}
CMC	11.7 ± 0.3^{b}	25.9 ± 0.8^{bc}	7.3 ± 0.4^{cd}	12.0 ± 0.3^{bcd}
MC	13.3 ± 0.1^{b}	28.1 ± 0.9^{bc}	10.5 ± 1.2^{b}	19.3 ± 1.5^{ab}
HPMC	8.6 ± 0.2^{b}	9.4 ± 0.6^{c}	7.8 ± 0.4^{cd}	5.7 ± 0.3^{d}
κ-Carrageenan	47.7 ± 8.0^{a}	70.6 ± 25.3^{a}	6.9 ± 0.8^{d}	12.7 ± 1.1^{bcd}
ί-Carrageenan	40.1 ± 1.4^{a}	55.5 ± 14.1^{ab}	37.5 ± 1.2^{a}	28.9 ± 10.0^{a}
λ-Carrageenan	11.8 ± 0.3^{b}	27.4 ± 3.0^{bc}	7.7 ± 0.4^{cd}	17.3 ± 2.1^{bc}

[1] Assays were performed in triplicate. Means ± SD values in the same column followed by different superscripts are significantly different ($P \leqslant 0.05$).
[2] 8 wt% normal rice starch (NRS) or waxy rice starch (WRS).

In general, these texture analysis data are consistent with the viscoelastic results (Tables 4 and 5) in which the more rigid starch–hydrocolloid mixed pastes with higher G' values gave higher gel hardness than the less rigid ones.

3.6. Microscopic observations

Light micrographs of NRS and WRS gels with and without hydrocolloids stained with iodine are shown in Figs. 1 and 2, respectively. Generally, it is known that amylose and/or amylopectin can and do leach from starch granules early in the pasting process (Shi & BeMiller, 2002; Tester & Morrison, 1990a). The microstructures of these starch–hydrocolloid gels were found upon cooling to phase-separate into a starch-rich disperse phase and a hydrocolloid-rich continuous phase, indicating thermodynamic incompatibility of starch and hydrocolloid molecules (Annable et al., 1994; Autio et al., 2002). All pastes contained very few, if any, identifiable granules or granule remnants. Amylose released from the granules appeared as small discrete particles within what is assumed to be a continuous hydrocolloid matrix. The presence of all hydrocolloids, except for ίC, also promoted amylopectin aggregation in the continuous phase so that a three-phase system containing amylose clusters, amylopectin clusters, and a solution of hydrocolloid molecules was formed. ί-Carrageenan did not enhance amylopectin aggregation in either the NRS (Fig. 1e) or the WRS (Fig. 2e) gels, possibly due to its association with amylopectin. This result is in good agreement with the texture analysis data (Table 8) in which the hardness and adhesiveness of the NRS–ίC and WRS–ίC gels were higher than those of the other gels, except for the NRS–κC gel.

In the case of NRS, addition of hydrocolloids resulted in a heterogeneous microstructure with amylose- and amylopectin-rich domains unevenly distributed in a hydrocolloid network matrix (Fig. 1a–f), whereas a more homogeneous structure in which very small amylose-rich domains uniformly dispersed in the gel matrix was observed in the NRS alone gel (Fig. 1g). This observation is congruent with the differences in the viscoelastic properties of these gels, for the NRS alone gel was found to be more structured and more solid-like, with a lower tan δ value, as compared to the NRS–hydrocolloid gels (Table 4). In contrast, the WRS gels seemed to be unaffected by the addition of hydrocolloids as evidenced by similar microstructures (Fig. 2a–g) and as well as similar tan δ values (Table 5).

When hydrocolloids are in an aqueous solution, water molecules are organized around the polysaccharide's hydroxyl groups, although some hydrocolloid hydroxyl groups may be hydrogen bonded to hydroxyl groups of adjacent hydrocolloid molecules rather than to water molecules. The main functional properties of hydrocolloids result from their relative abilities to organize water and/or to form networks, dividing them into two categories: thickeners only and gelling agents. Thickener molecules move about randomly and exhibit little interaction with each other, whereas gelling molecules form junction zones and create a three-dimensional network structure (Whistler & BeMiller, 1997). The results obtained in this work appear to demonstrate that the non-gelling hydrocolloids enhanced the viscosity of the starch pastes, whereas the gelling hydrocolloids enhanced the hardness, adhesiveness, and viscoelastic properties of the pastes.

4. Conclusions

Addition of various cellulose derivatives and carrageenans to NRS or WRS suspensions resulted in an increase in pasting temperatures and peak and final viscosities in the following decreasing order: MC > CMC > HPMC and λC > ίC > κC. SP and SOL of NRS increased significantly when hydrocolloids were added. SP and SOL of WRS were much less affected by the addition of hydrocolloids. Dynamic rheological measurements revealed no effect of the hydrocolloids, except for κC, on G', G'', or η^*, but the tan δ values of the NRS pastes were increased by the addition of hydrocolloids, indicating that the pastes were less structured and less solid-like than the control paste. On the contrary, the hydrocolloids had no effect on the tan δ values of WRS pastes, indicating that their structures were similar to those of the control. Steady shear tests indicated that all the hydrocolloids, except for HPMC, increased $\eta_{a,100}$ and K values of both starches with trends

similar to those found during pasting, whereas the opposite trend was observed for *n* values. Textural tests demonstrated that the added κC or íC significantly increased hardness and adhesiveness of the NRS pastes, whereas the other hydrocolloids did not significantly affect these textural properties. A similar result was also observed in the case of WRS, except for κC, which significantly decreased the hardness of the paste. Microscopic examination showed a phase-separate microstructure of the starch–hydrocolloid pastes in which starch-rich domains were dispersed in a continuous hydrocolloid matrix.

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