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Influence of pH and temperature on the rheological properties of aqueous dispersions of starch–sodium palmitate complexes

Jeffrey A. Byars[a,*,1], George F. Fanta[b], James A. Kenar[a], Frederick C. Felker[a]

[a] Functional Foods Research Unit, National Center for Agricultural Utilization Research, Agricultural Research Service, United States Department of Agriculture, 1815 N University St., Peoria, IL 61604, USA
[b] Plant Polymer Research Unit, National Center for Agricultural Utilization Research, Agricultural Research Service, United States Department of Agriculture, 1815 N University St., Peoria, IL 61604, USA

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ABSTRACT

Aqueous dispersions of high-amylose corn starch were steam jet cooked and blended with aqueous solutions of sodium palmitate to form amylose inclusion complexes. The rheology of dispersions of these complexes was examined. Acetic acid was added to reduce the pH, converting complexed sodium palmitate to palmitic acid. Associations of the complexed palmitic acid and reduced electrostatic repulsion resulted in a sharp increase in the linear viscoelastic properties of the materials. Further reduction of the pH led to precipitation of palmitic acid complexes and a decrease in the gel strength. Temperature ramps showed a sharp, reversible decrease in the elastic modulus. The temperature at which this decrease occurred depended on both the concentration and pH.

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1. Introduction

In a series of studies aimed at producing new starch-based composites based on amylose inclusion complexes, we have used steam jet cooking as an inexpensive, large-scale processing method. The high-temperature and high-shear conditions of excess steam jet cooking (Klem & Brogley, 1981) substantially solubilize granular starch and reduce its molecular weight (Byars, 2003; Dintzis & Fanta, 1996). When cooled, complexes formed from dispersions of jet-cooked starch and palmitic or oleic acid formed insoluble spherulites, the size and morphology of which depended on the fatty acid and the cooling rate (Fanta, Felker, Shrogren, & Salch, 2008), and concentrated dispersions of the spherulites formed smooth, flowable pastes with flow properties similar to those of shortenings (Byars, Fanta, & Felker, 2009). When composites containing the complexes and oil were quickly cooled, the inclusion complexes formed submicron particles that allowed very high oil concentrations to be entrapped in stable, aqueous systems (Byars, Fanta, & Felker, 2011; Fanta, Felker, Byars, Kenar, & Shogren, 2009). When helical inclusion complexes were formed between amylose and sodium palmitate, spherulites did not form upon cooling, but the starch instead remained soluble and formed low-viscosity liquids or gels depending on the concentration (Fanta, Kenar, Byars, Felker, & Shogren, 2010).

Amylose inclusion complexes have been studied extensively, and their preparation and properties have recently been reviewed (Putseys, Lamberts, & Delcour, 2010). Applications of the complexes include their effect on the pasting of starch (Blazek & Copeland, 2009; Blazek, Gilbert, & Copeland, 2011; Mira, Eliasson, & Persson, 2005; Raphaelides & Georgiadis, 2008), retrogradation (Keetels, van Vliet, Jurgens, & Walstra, 1996), and protection of oxygen sensitive molecules (Lalush, Bar, Zakaria, Eichler, & Shimoni, 2005; Lay Ma, Floros, & Ziegler, 2011; Yang, Gu, & Zhang, 2009). Although a number of studies have considered the effects of amylose–lipid complexes on starch gelatinization and gelation, only a few previous studies have focused on the rheology of materials containing inclusion complexes. Biliaderis and Tonogai (1991) showed that lipid complexes increased the strength of concentrated gels of rice and wheat starch, and inhibited staling. Chien, Lien, and Shoemaker (1999) also showed that amylose–lauric acid complexes increased the strength of concentrated rice starch gels, and that the complexes led to an increase in gel strength upon storage. The effect

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[*] Corresponding author. Tel.: +1 309 681 6330; fax: +1 309 681 6685.
E-mail address: jeffrey.byars@ars.usda.gov (J.A. Byars).

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of temperature on the rheology of gels containing starch–lipid complexes was studied by Kim, Eliasson, and Larsson (1992) and Eliasson and Kim (1995). They observed a sharp decrease in the storage modulus and increase in the phase angle upon heating. The transition temperature depended on the type of starch, the chain length and head group of the lipid, and the salt concentration. These differences were attributed to dissociation of the amylose–lipid complex. To the best of our knowledge, the only other previous studies on the rheology of systems consisting primarily of amylose–lipid complexes were for systems containing starch and fatty acids (Raphaelides, 1992a) and amylose and potassium fatty acid salts (Raphaelides, 1992b). The addition of fatty acids was shown to reduce the viscosity of amylose solutions at high shear rates at pH 12, and the rigidity of amylose gels increased with the addition of fatty acid salts. Fanta et al. (2010) observed that the viscosity of a dispersion of jet-cooked high-amylose corn starch with sodium palmitate increased and then decreased sharply over a narrow range of pH as it was titrated with hydrochloric acid. The goal of the current study is to examine in more detail this system to understand the effects of concentration, pH and temperature on the rheological properties of the dispersions.

2. Materials and methods

2.1. Materials

High-amylose corn starch (AmyloGel 03003, containing approximately 70% apparent amylose) was a product of Cargill (Minneapolis, MN). Percent moisture was calculated from weight loss after drying at 100 °C under vacuum. All weights of starch and starch–sodium palmitate complexes are given on a dry weight basis. Sodium palmitate (98.5%) was purchased from Sigma (St. Louis, MO). Acetic acid (Certified ACS grade) was purchased from Fisher Scientific (Waltham, MA).

2.2. Preparation of starch–sodium palmitate complexes by steam jet cooking

High-amylose corn starch (150.0 g) was dispersed in 2700 mL of deionized water, and the slurry was passed through a Penick & Ford laboratory model steam jet cooker (Penford Corp., Englewood, CO) operating under excess steam conditions (Klem & Brogley, 1981). The temperature in the hydroheater was 140 °C, the steam back pressure was 380 kPa (40 psig) and the steam line pressure from the boiler was 550 kPa (65 psig). The flow rate of the dispersion through the jet cooker was about 1 L/min. The hot, jet-cooked starch dispersion was collected in a tared Waring blender (Waring Products Division, New Hartford, CT) that was previously heated with 100 °C water from the jet cooker, and the weight of jet-cooked dispersion in the container was determined. The jet cooker was flushed with excess water to maximize the amount of starch collected. The weight of hot dispersion in the blender was 3369 g and the percent starch solids in the cooked dispersion were determined by freeze drying a weighed amount of the hot dispersion. From these values, it was determined that 97% of the initial weight of starch was collected in the jet-cooked dispersion.

Sodium palmitate (7.88 g) equal to 7.5% of the weight of amylose in the starch sample (assuming a 70:30 weight ratio of amylose:amylopectin) was dissolved in 300 mL of water at about 95 °C, and the resulting clear solution was added to the hot starch dispersion immediately after it was collected from the jet cooker. The resulting dispersion was slowly stirred for about 2 min, and the dispersion was then transferred to a 4 L beaker and cooled in an ice-water bath to 25 °C. The cooled solution was then centrifuged for 1 h at 10,000 rpm, relative centrifugal force of approximately 17,000 × g, using a Beckman (Palo Alto, CA) J2-21 ME centrifuge equipped with a JA-10 rotor. The supernatant was then freeze dried to yield 146 g of starch–sodium palmitate complex that was stored for later experiments.

2.3. Preparation of starch–sodium palmitate gels

Dispersions containing 2, 3 or 4 wt% of the freeze-dried starch–sodium palmitate complex in water were prepared by adding deionized water to the complex (0.90, 1.35 or 1.80 g, respectively) to give a total weight (complex + water) of 45.0 g. The mixtures were stirred and heated to 80 °C to dissolve the complex. While at 80 °C, 0.1 M acetic acid (200–1800 μL) was added to the solution to adjust the pH. The mixture was then stirred at 80 °C for an additional 10–20 min to allow sufficient mixing and to obtain a homogeneous solution. The solutions were then removed from the heat and allowed to cool slowly to ambient temperature. After cooling, the pH was measured (Orion 8235BN electrode and ThermoOrion 370 meter, Thermo Fisher Scientific, Waltham, MA). Complete dissolution of the freeze-dried complexes was confirmed by the absence of any particulate material when viewed by bright field or phase contrast optics using a Zeiss Axioskop 50 light microscope (Carl Zeiss, Inc., Thornwood, NY).

2.4. Rheological property measurements

Measurements in small amplitude oscillatory shear flow were conducted on an ARES LS1 (TA Instruments, New Castle, DE) controlled strain fluids rheometer. Tests were performed with a 25 or 50 mm diameter parallel plate geometry at strains within the linear viscoelastic region for all samples. A Peltier plate was used to maintain the temperature at 25.0 ± 0.1 °C or to conduct temperature ramps at 1 °C/min. Sample edges were coated with mineral oil and humidity covers were used to prevent drying of the samples.

2.5. Differential scanning calorimetry

Thermal properties were determined using a TA instruments (New Castle, DE) model Q2000 differential scanning calorimeter (DSC) calibrated against an indium standard (156.6 °C, 28.86 J/g). Data was collected and analyzed with TA Universal Thermal Analysis 2000 V3.9A software. Samples were prepared at 20–25 wt% (dry weight basis) of sample in Millipore water using high volume stainless steel DSC pans. The pans were hermetically sealed and allowed to equilibrate at least 4 h before analysis. The sealed DSC samples were run and referenced against an identical stainless steel pan containing water. The samples were analyzed using modulation (±0.40 °C every 40 s) at a heating rate of 5.0 °C/min while heating from 5 to 140 °C.

3. Results and discussion

Excess steam jet cooking is an effective method of rapidly producing large quantities of amylose helical inclusion complexes. Fatty acids can be passed through the jet cooker with the starch to form the complexes in a single step, but Fanta et al. (2010) showed that fatty acid salts caused foaming in the jet cooker which interfered with heat transfer to the starch granules, so the fatty acid salts must be added to the hot, cooked starch dispersion. A sodium palmitate concentration of 6% based on apparent amylose content resulted in cloudiness upon standing, suggesting retrogradation of uncomplexed amylose. However, at the sodium palmitate concentration of 7.5% based on apparent amylose used in this study there was no visual or microscopic evidence of retrogradation and a low viscosity liquid was obtained. Although Fanta et al. (2010) studied jet-cooked materials that were not dried, in this work we

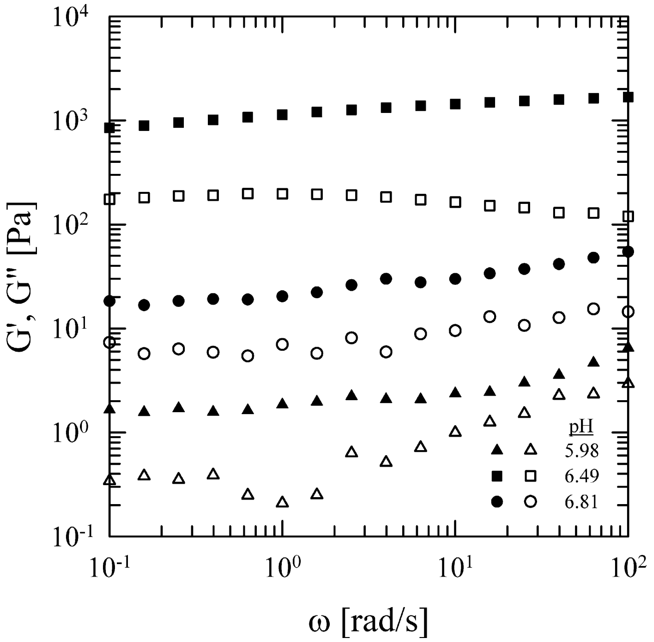

Fig. 1. Storage modulus, G' (solid symbols) and loss modulus, G'' (open symbols) for samples with 3% starch–sodium palmitate complexes.

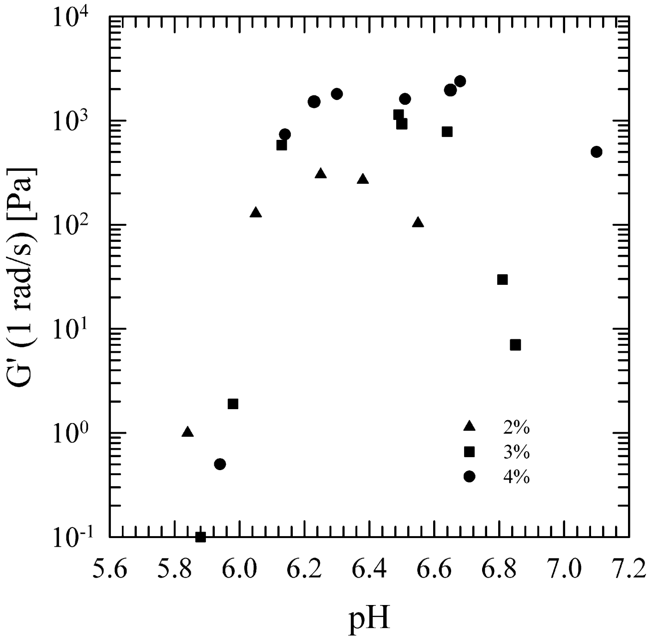

Fig. 2. Storage modulus at 1 rad/s for different starch–sodium palmitate levels as a function of pH.

freeze-dried and then reconstituted the starch–sodium palmitate complexes. Variations in the amount of starch recovered from the jet cooker and the total volume of recovered sample could affect both the starch concentration and the ratio of sodium palmitate to amylose. All of the starch–sodium palmitate complexes used in this study were therefore prepared in a single jet-cooking experiment to ensure uniform properties across the range of samples studied. After adding the acid to the sample at 80 °C, samples were allowed to stand for at least 2 h to cool to room temperature and form gels. The reported values were then measured, and subsequent measurements showed that both the pH and rheological properties did not change significantly over at least 24 h. The addition of the acetic acid not only changed the pH, but also resulted in the formation of sodium acetate in solution as the complexed sodium palmitate salt was neutralized by the acetic acid. Fanta et al. (2010) showed that the addition of sodium chloride influenced the viscosity of starch–sodium palmitate complex dispersions, but the low salt concentrations present in the current study are not expected to have a significant effect on the rheology. The maximum concentration of sodium acetate in the current study was 0.004 M, whereas Fanta et al. (2010) did not observe an increase in the viscosity for sodium chloride concentrations as high as 0.011 M. The effect on the rheology would also be less because of the reduced ionization of sodium acetate compared to sodium chloride.

The linear viscoelastic properties of solutions containing 3 wt% complexed starch at different pH values are shown in Fig. 1. Each sample formed a gel, as indicated by the weak frequency dependence of the moduli, and the fact that the storage modulus (G') was higher than the loss modulus (G'') for all frequencies, although the strength of the gel as indicated by the ratio G'/G'' also depended on pH. Changing the pH over a narrow range led to dramatic changes in the gel properties as the interactions between the amylose molecules were altered. At pH > 6.8, the amylose complexes remained water soluble due to electrostatic repulsion keeping the polymer in an extended conformation (Karlberg, Piculell, & Huang, 2007), and intermolecular entanglements may exist. As acid was added, complexed sodium palmitate was partially converted to palmitic acid. The electrostatic repulsion was therefore decreased, and junction zones could form between converted complexes or between converted complexes and amylopectin (Klucinec & Thompson, 1999, 2002). At intermediate pH values, a balance exists between these forces and very strong gels can be formed. However, as the pH was further decreased, most of the complexes contained palmitic acid, which formed insoluble aggregates, as was found when palmitic acid was used to complex amylose (Fanta et al., 2008).

Similar results were obtained for dispersions with 2 and 4 wt% complexed starch, and the results are summarized in Fig. 2. The value of the storage modulus at a frequency of 1 rad/s is presented as a characteristic measure of the gel strength for each sample. Samples containing 2% complexes followed the same trend as samples with 3% complexes, although increased gel strength was not observed over as wide a range of pH values, and the peak value of G' was shifted to lower pH. For samples with 4% complexes, the maximum value of G' was only slightly higher than for 3%, although high G' values were observed over a much wider range of pH, suggesting that these samples were sufficiently concentrated for entanglements to affect the gel properties. At low pH, the modulus values decreased sharply for all samples as the pH was reduced below 6.1. At this pH, most of the sodium palmitate complexes have been converted to palmitic acid complexes, which then precipitated from solution and no longer contributed to the modulus value of the gel.

Above pH 6, the gels that formed were thermoreversible. Upon heating, the storage modulus initially decreased slowly, followed by a sharp drop, as shown in Fig. 3 for samples with 4% starch complexes. When cooled, the storage modulus increased, usually returning to its initial value as for the sample at pH 6.51; however, in some cases the cooled sample had slightly lower G' values, as was the case for the sample at pH 6.87. In all cases, the shape of the cooling followed that of the heating curve. Eliasson and Kim (1995) observed similar decreases in the elastic modulus for gels containing a wide range of amylose–lipid complexes. They attributed the decrease to dissociation of the amylose inclusion complexes, but that does not seem to account for our results. A DSC scan of the freeze-dried starch–sodium palmitate complexes is shown in Fig. 4. A large peak was observed at 98.1 °C ($\Delta H = 26.5$ J/g), and a smaller peak was at 118.5 °C ($\Delta H = 0.9$ J/g). These values are in good agreement with those reported by Tufvesson, Wahlgren, and Eliasson

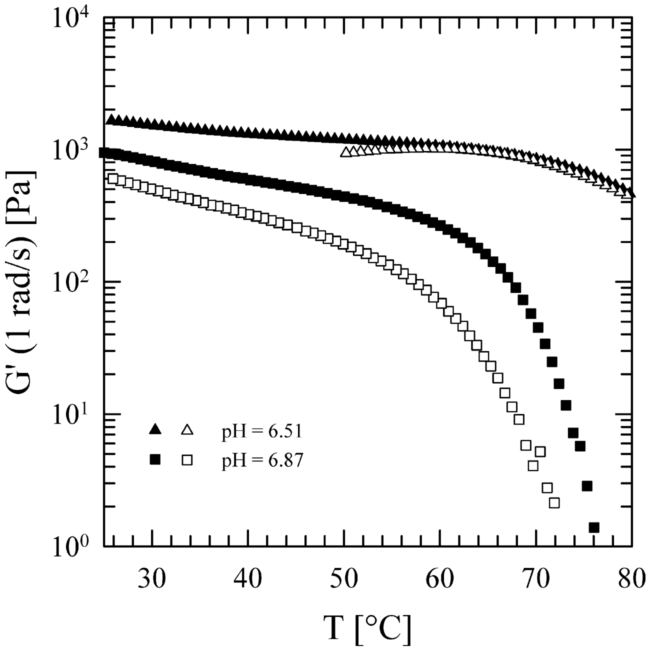

Fig. 3. Storage modulus at 1 rad/s for samples with 4% starch–sodium palmitate complexes during heating (solid symbols) and cooling (open symbols).

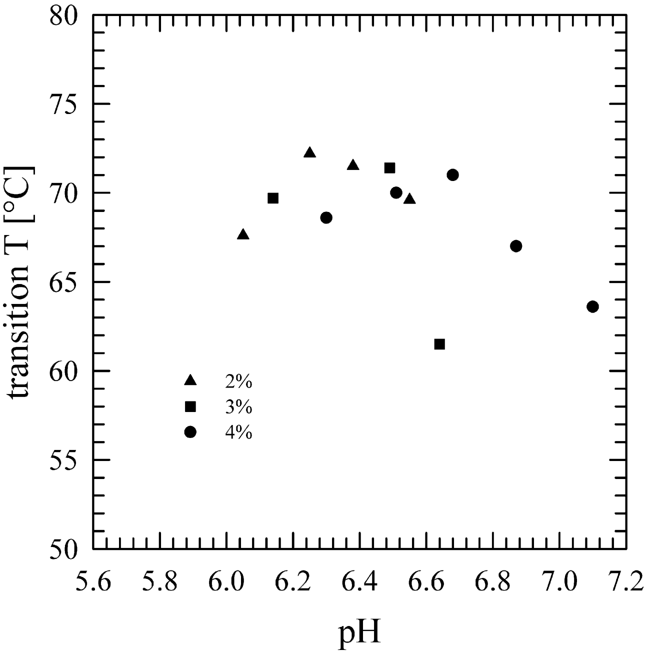

Fig. 5. Transition temperature during heating as a function of pH.

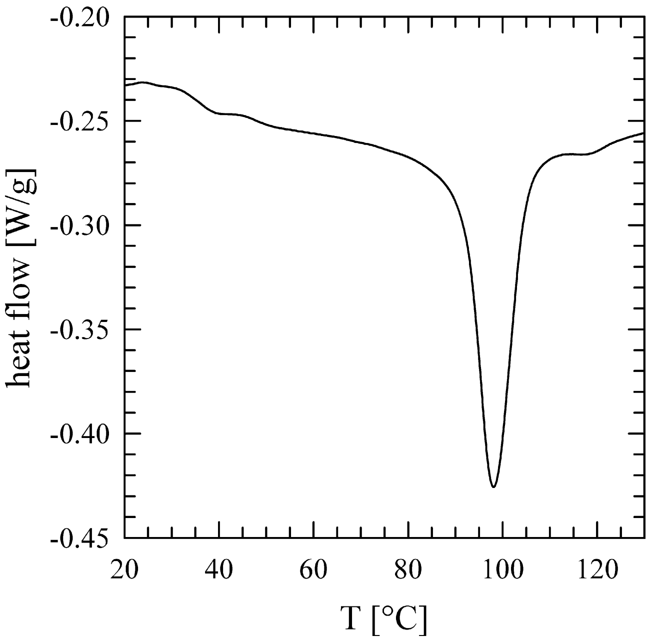

Fig. 4. DSC scan of freeze-dried starch–sodium palmitate complexes.

(2003) for the dissociation of amorphous complexes (form I) and the melting of crystallized complexes (form II). The onset of the dissociation peak was at 91.4 °C, which was still above the temperature where the modulus values of the gels decreased. The DSC results also show that neither the jet cooking procedure nor the freeze-drying of the complexes resulted in the formation of a significant fraction of crystallized complexes.

Eliasson and Kim (1995) calculated a transition temperature for the decrease in G' based on the value of the phase angle, δ, where $\tan\delta = G''/G'$. They observed a large change in the phase angle over a narrow temperature range, with nearly constant values at low and high temperatures. The temperature at the midpoint of this range was defined as the transition temperature. That method could not be used for the current study, since no plateau value for either G' or δ was reached at high temperatures. Their samples contained 5% swollen starch granules, which may have provided a small viscosity component even when the complexes no longer contributed to the gel strength, whereas the current samples contained only a small amount of amylopectin in addition to the complexed amylose. Instead, the transition temperature was estimated by extrapolating the G' values at low and high temperatures, where $\log G'$ vs. T was linear, and calculating the intersection of the two lines. These results are summarized in Fig. 5. For each concentration, the maximum transition temperature was 71–72 °C, and the pH for the maximum value shifted to higher pH with increasing concentration. The same complexes were used for all of the samples, so the range of transition temperatures is further evidence that the transition is not due to dissociation of the amylose–sodium palmitate (or amylose–palmitic acid) complex.

As was seen in Fig. 3, the extent of the decrease in G' was not the same for all samples. The storage modulus at 80 °C was between 1% and 35% of the value at 25 °C, and the extent of the decrease depended strongly on pH, as shown in Fig. 6. Although the results in Fig. 5 are not sufficient to determine precisely the pH of the maximum transition temperature, the shift in the curves to higher pH in Fig. 6 with increasing concentration is similar to the shift of the maximum transition temperature with pH. Although it is reasonable that samples with higher transition temperatures may not have their modulus values decrease as much if they are only heated slightly above their transition, for each concentration the sample at the lowest pH had a transition temperature below the maximum, yet still had a relatively higher modulus value at 80 °C. These results suggest that there is a qualitative change in the type of junction zones formed in the gels with changing pH. At pH where the complexes remained highly charged, intermolecular interactions were primarily due to hydrogen bonding, which can be disrupted at elevated temperatures. As the pH was decreased, reduced electrostatic repulsion and junction zones between amylose–palmitic acid complexes not only strengthened the gel as shown above, but also led to more temperature-stable gels.

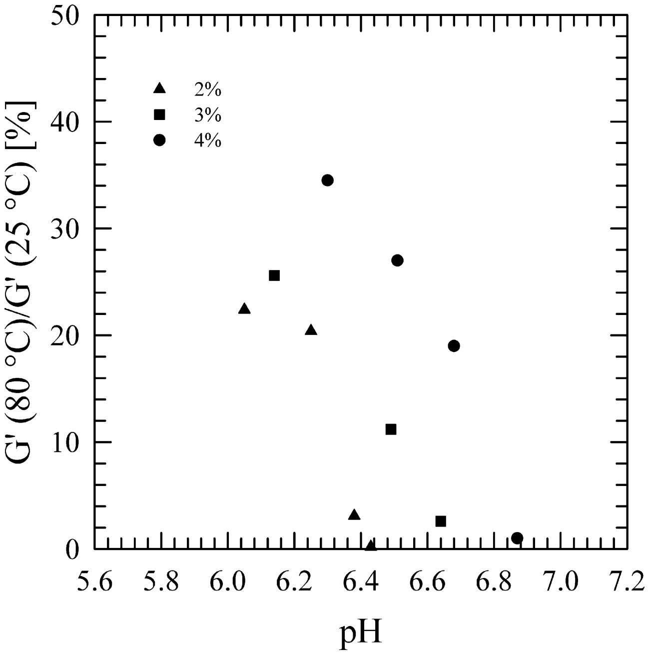

Fig. 6. Ratio of the storage modulus at 1 rad/s at 80–25 °C as a function of pH.

4. Conclusions

Aqueous dispersions of high-amylose corn starch were steam jet cooked and blended with aqueous solutions of sodium palmitate to form amylose inclusion complexes. Dispersions of the starch–sodium palmitate complexes were prepared at 2, 3 and 4 wt% to examine their rheological properties. As acetic acid was added to lower the pH, the samples formed gels over a narrow pH range for 2 and 3% samples, and a broader range for samples with 4% complexes. In each case, the maximum of the storage modulus was observed at about pH 6.3. Below pH 6.1, the complexes precipitated out of solution, and the modulus values for all concentrations dropped sharply with pH. These results were attributed to changes in the intermolecular interactions as the charge on the amylose complexes changed. At high pH, electrostatic repulsion kept molecules in solution and fully extended, and only the 4% sample was sufficiently concentrated for entanglements to affect the rheology significantly. As the pH was reduced, the amount of electrostatic repulsion decreased, and junction zones could form between molecules, leading to the gel structure. The junction zones resulted from interactions between complexes in which the sodium palmitate had been converted to palmitic acid. At the lowest pH values, most of the sodium palmitate had been converted to palmitic acid, so the interactions between complexes became even stronger, leading to the formation of insoluble particles and a decrease in the gel strength. Upon heating, the modulus values dropped rapidly at a transition temperature near 70 °C, but recovered upon cooling. The transition temperature depended on pH, and was shifted to higher pH values for higher concentrations. The extent of the drop of the modulus values depended on pH, due to changes of the gel structure with pH. The modulus values of samples at lower pH with interactions primarily due to junction zones between palmitic acid complexes decreased much less than samples at higher pH with interactions primarily due to hydrogen bonding. The gelling properties of these materials suggest practical applications as thickeners and as dispersants for lipids in foods, lotions and water-based lubricants.

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