

Effects of adding acids before and after gelatinization on the viscoelasticity of cornstarch pastes

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Abstract

Effects of sour substances (citric acid, acetic acid, lactic acid, malic acid, tartaric acid, and ascorbic acid) on the rheological properties of cornstarch pastes were studied by steady shear and dynamic oscillatory viscoelasticity measurements, intrinsic viscosity measurements, and microscopic observation. The pH of cornstarch dispersion was adjusted between 6.0 and 3.0. The viscosity of the pastes was increased by lowering the pH (between 5.5 and 3.6), while the viscosity of samples with pH below 3.5 decreased further than that of the control (pH = 6.3). Hydrolysis of amylose and amylopectin chains occurred by adding sour substances, and led to the decrease in the viscosity at lower pH. However, adding a small amount of sour substances promoted leaching out of amylose and amylopectin chains, and led to the increase in the viscosity. Similar tendencies were observed by adding various sour substances used to adjust pH.

No decrease in the viscoelasticity was observed for cornstarch pastes by adding acids at 25 °C after gelatinization.

Keywords: cornstarch, viscoelasticity, pH, citric acid, acetic acid, lactic acid, malic acid, tartaric acid, ascorbic acid

1. Introduction

Starch is present in the form of granules, which are not soluble in water. The main constituents of starch are amylose and amylopectin, which consist of α -D-glucose residues by α -(1→4) and α -(1→6) linkages, respectively, that are large polysaccharide molecules. When starch is heated in water at a higher temperature (about 80 °C), the starch granules swell and amylose can leach out from the granules. Amylopectin can also leach out from the granules, though it leaches out more slowly than amylose. In consequence, starch granules rupture and the starch suspension becomes a viscous paste. This process is known as gelatinization. Starch has been widely used in the food industry as a thickener, a stabilizer or a gelling ingredient using this property.

Foods contain intricate mixtures of tastes, and products made of starch are no exception. The tastes are mainly controlled by seasoning substances, some of which are known to have effects on the physical properties of starch pastes and gels.

There are many reports concerning the effects of taste substances on gelatinization and retrogradation of starch (Abu-Jdayil, Azzam, & Al-Malah, 2001; Ahmad, & Williams, 1999; Campbell, & Briant, 1957; Cheer, & Lelievre, 1983; Chungcharoen, & Lund, 1987; D' Appolonia, 1972; Eliasson, 1992; Evageliou,

Richardson, & Morris, 2000; Evans, & Haisman, 1982; Katsuta, Miura, & Nishimura, 1992; Kim, & Walker, 1992; Kohyama, & Nishinari, 1991; Maaurf, Che Man, Asbi, Junainah, & Kennedy, 2001; Perry, & Donald, 2002; Plata-Oviedo, & Camargo, 1998; Spies, & Hosney, 1982; Sriburi, Hill, & Mitchell, 1999; Vallés-Pámies, Barclay, Hill, Mitchell, Paterson, & Blanshard, 1997; Yamada, Morimoto, & Hisamatsu, 1986), while there are few reports concerning the viscoelasticity of starch (Abu-Jdayil, Azzam, & Al-Malah, 2001; Ahmad, & Williams, 1999; Evageliou, Richardson, & Morris, 2000; Vallés-Pámies, Barclay, Hill, Mitchell, Paterson, & Blanshard, 1997). It is necessary to investigate the effects of seasonings on the viscoelasticity of starch since it may lead to understanding how to control the viscosity of starch products.

In this study, cornstarch and sour substances (various acids) were selected. Cornstarch is the most widely used starch in the food industry. Since starch is hydrolyzed by acid, aqueous hydrochloric acid was used to make modified starch by compulsorily causing hydrolysis of amylose and amylopectin chains (glucose chains) (Chamberlain, & Rao, 2000; Li, Vasanthan, Rossnagel, & Hoover, 2001; Wurzburg, 1995). However, sour substances are included in many foods and are added to many foods as an acidulent or a preservative.

Our objective was to investigate how sour seasonings affect the physical properties of starch pastes and contribute to the effect of increasing the viscosity for starch pastes. Acids such as citric acid, acetic acid, lactic acid, malic acid, tartaric acid, and ascorbic acid were used to lower the pH as a model for actual starch food products. Citric acid is contained in citrus fruits and is added to foods as a sour seasoning. It is also important as a sour basic substance for difference tests on sensory evaluation. Acetic acid is the main sour component of vinegar. Lactic acid is included in cheese and

yogurt. Malic acid and tartaric acid are in many kinds of fruits and are used as acidulents. Ascorbic acid is an important source of vitamin C; it is also contained in many kinds of vegetables and fruits and is used as an antioxidant.

2. Materials and methods

2.1 Materials

A cornstarch sample (cornstarch Y) was kindly provided by Sanwa Starch Co. Ltd. (Nara, Japan). The concentration of cornstarch was fixed at 3.00 wt%; this is the concentration generally used for thickeners in sauces or soups. The moisture content of the cornstarch was determined to be 13.4 % by drying it at 130 °C in atmospheric pressure. Citric acid (anhydrous), acetic acid, DL-lactic acid, DL-malic acid, L(+)-tartaric acid, L(+)-ascorbic acid, and dimethyl sulfoxide were of reagent grades. The pH was adjusted between 6.0 and 2.4. Sorbic acid potassium salt (0.05 wt%) was used to prevent microbial growth.

2.2 Preparation of cornstarch pastes

Powders of cornstarch were dispersed in distilled water or in acid aqueous solutions with pH adjusted beforehand. A control sample of pH6.3 was made of distilled water and cornstarch. Cornstarch dispersions were stirred at 200 rpm for 30 min at 25 °C, then the dispersions were heated up to 97 °C, stirring at the same rate and maintained at 97 °C for 60 min. Then the hot dispersions were poured into glass vessels (140 mL volume) and cooled in a water bath (5 L volume) at 25 °C for 60 min equipped

with a temperature regulator, stirring with magnetic stirrers. Other starch pastes were also prepared; after heating and cooling the starch dispersions, i.e., after gelatinization of starch, pH was adjusted. The final concentration of cornstarch was 3.0 wt%.

2.3. Steady shear and dynamic oscillatory measurements

Steady shear and dynamic oscillatory measurements of starch pastes were carried out using a Fluids Spectrometer RFS II (Rheometrics Co. Ltd., New Jersey, U.S.A.) with a 50 mm diameter plate geometry. The gap was adjusted to 1.00mm. All measurements were made at 25.0 °C. From these measurements, shear viscosity ($\eta_{\dot{\gamma}}$), storage modulus (G'), loss modulus (G''), and complex viscosity ($|\eta^*|_{\omega} = \sqrt{G'^2 + G''^2} / \omega$) were obtained. Strain for dynamic oscillatory measurements was chosen in the plateau range of each sample on the strain dependence test.

2.4. Microscopic observations

Microscopic observations were made using a light microscope, OPTIPHOTO (Nikon Co. Ltd., Tokyo, Japan) equipped with 2D-color CCD (1280 × 960 pixels). Unheated cornstarch dispersions and heated cornstarch pastes were observed. Pastes prepared by the method mentioned above were diluted with 2 times of distilled water. Granules of cornstarch were dyed with a 10 mM iodine solution.

2.5. Intrinsic viscosity measurements

Cornstarch (3.0 wt%) was dispersed in distilled water or acids aqueous solutions with the pH adjusted beforehand. The dispersions were heated in a water bath at 97 °C for 60 min stirring with magnetic stirrers, then cooled in a water bath at 25 °C for 60 min stirring. Dimethyl sulfoxide (DMSO) was then added to the dispersions so that the ratio of DMSO to water became 90:10 (vol). Starch can be dissolved completely in 90 vol% DMSO aqueous solutions (Jackson, 1991). The solutions were heated and cooled again using the same methods mentioned above. The concentration of starch became 0.28 wt%. The solutions were serially diluted by 90 vol% DMSO aqueous solution to a final concentration of 0.06 wt%. Intrinsic viscosity measurements were made using an Ubbelohde type capillary viscometer at 40.0 ± 0.01 °C. Intrinsic viscosity ($[\eta]$) was calculated from the Huggins equation (Huggins, 1942):

$$\eta_i / c = [\eta](1+k[\eta]c+\dots)$$

$$\eta_i = (\eta - \eta_0) / \eta_0$$

where η_0 is the viscosity of the solvent, η is the viscosity of the solution, η_i is the relative viscosity increment, c is the concentration of the solute, and k is the Huggins constant.

3. Results and discussion

3.1 Steady shear measurements for starch pastes adding acids before gelatinization

It was found that all samples showed typical shear-thinning behaviors, and shear viscosity ($\eta_{\dot{\gamma}}$) was affected by both shear rate ($\dot{\gamma}$) and pH from steady shear measurements (data not shown).

The pH dependence of the shear viscosity at $\dot{\gamma} = 10 \text{ s}^{-1}$ (η_{10}), is shown in Fig.

1. The pH was adjusted by adding various acids before gelatinization. η_{10} decreased by lowering the pH to around 5.6 and increased inversely at $3.6 < \text{pH} < 5.6$. By lowering the pH furthermore, η_{10} decreased again. The values of η_{10} , where the pH was above 5.5 and below 3.6, were less than that of the control, and the values of η_{10} , where the pH is between 5.5 and 3.6, were greater than that of the control.

The pH at which the viscosity changed was only slightly dependent on the types of acids, and the general tendency mentioned above was not dependent on the types of acids. In other words, the viscosity of the pastes is a function of pH and does not depend on types of acids. The change of the viscosity was induced by the changes in the size of starch granules and the number of amylose and amylopectin chains (glucose chains), which were caused by pH change.

Many researchers reported that the viscosity of starch pastes decreased in the presence of acids (Campbell, & Briant, 1957; D' Appolonia, 1972; Sriburi, Hill, & Mitchell, 1999; Sriburi, & Hill, 2000; Vallés-Pámies, Barclay, Hill, Mitchell, Paterson, & Blanshard, 1997). Yamada, Morimoto, & Hisamatsu (1986) reported that the viscosity of pastes for potato starch decreased by adjusting the pH with citric acid or acetic acid, while that for cornstarch was not affected by acids. However, an increase in the viscosity for starch pastes by adding acids has not yet been reported to our knowledge.

3.2. Microscopic observation

The viscosity of starch pastes is affected by the size of starch granules and the number of glucose chains leaching out from starch granules. Microscopic photographs of the starch granules, the pH of which was adjusted by adding citric acid before gelatinization, are lined on the left in Fig. 2.

The granule sizes and shapes before heating for all samples with adjusted pH were similar to those of the control (Fig. 2, top left). There were no differences among all samples (data not shown).

The heated cornstarch granules were swollen and partly ruptured, and then glucose chains were leached out from starch granules. The granule sizes of the samples at pH6.0, 5.0, and 4.0 with the pH adjusted before gelatinization were almost the same as those of the control. However, the number of glucose chains at pH5.0 and 4.0 (blue region in Fig. 2) was apparently more than that of the control. Therefore, there are many entanglements of glucose chains as compared with those of the control, and these networks of glucose chains lead to higher viscosities. Thus, the viscosity of starch samples, where the pH is between 5.5 and 3.6, was greater than that of the control. Many small granules were observed for the sample with pH = 3.5 as compared with the control and the samples at pH6.0, 5.0, and 4.0. Almost all granules were broken at pH3.0. Decreases in the viscosity by lowering pH below 3.6 may be due to the collapse of many starch granules or the hydrolysis of glucose chains by adding acids.

3.3. Intrinsic viscosity measurement

If the hydrolysis of glucose chains were to occur by adding acid, the degree of polymerization (DP) for glucose chains should decrease. Intrinsic viscosity ($[\eta]$) was

then measured to confirm that the hydrolysis of glucose chains did occur. Since $[\eta]$ depends on DP , the size of glucose chains can be estimated from changes in $[\eta]$. Fig. 3 shows the results of intrinsic viscosity measurements. The pH of the x-axis is equivalent to the pH in the preparation for cornstarch pastes. The pH was adjusted by adding citric acid before gelatinization. $[\eta]$ decreased slightly with acidification at $pH \geq 4.0$. This decrease indicates that few glucose chains were hydrolyzed by adding acid. However, increases in the viscosity were observed, where the pH is between 5.5 and 3.6 as shown in Fig. 1. It is thought that acids enter the starch granules, causing the hydrolysis of glucose chains inside starch granules, which then makes many glucose chains leach out into the starch pastes. Therefore, the entanglements of glucose chains increase further than those of the control. $[\eta]$ decreased sharply at $pH < 4.0$. This indicates that hydrolysis of glucose chains occurred by adding acid and DP decreased further than that at $pH \geq 4.0$. Decreases in the viscosity were observed, where the pH is below 3.5, since the entanglements of glucose chains were less than those at $pH \geq 4.0$. Precipitation of starch in starch pastes occurred at $pH < 2.7$ (Fig. 1 and 3). The precipitation of starch is thought to be caused by many glucose chains with low DP in starch pastes. Burchard (1963) and Pfannemüller (1971) reported that the solubility of amylose with various DP increased considerably with shorter and longer molecules. Gidley, & Bulpin (1989) also reported that amylose with DP between 40 and 660 were found to precipitate, although the precipitation depended on the concentration of amylose.

3.4. Dynamic oscillatory measurements for starch pastes adding acids before gelatinization

Fig. 4 illustrates the frequency (ω) dependence of storage modulus (G') (Fig. 4A) and loss modulus (G'') (Fig. 4B) of cornstarch pastes with or without adjusting the pH by adding tartaric acid before gelatinization. The samples of the control (pH = 6.3) and with pH6.0 showed behaviors known for concentrated flexible polymer solutions, i.e., G' is less than G'' at lower ω and G' is greater than G'' at higher ω . The values of G' and G'' for a paste with pH6.0 were a little greater than those of the control. The values of G' were greater than those of G'' at all ω examined and G' and G'' increased with increasing ω for pastes with pH5.0 and 4.0. This behavior resembles that for a weak gel, which consists of entangled networks formed by starch granules and glucose chains that leach out from starch granules. The paste behaved as a concentrated solution where the pH is 3.0. The values of G' and G'' were less than those of the control at all ω examined. This suggests that the size of starch granules and DP for glucose chains both decreased.

Complex viscosity ($|\eta^*|_\omega$) of 3.0 wt% cornstarch pastes with adjusted pH by various acids showed a similar tendency to $\eta_{\dot{\gamma}}$. Fig. 5 shows pH dependence of $|\eta^*|_\omega$ at $\omega = 10 \text{ rad s}^{-1}$ ($|\eta^*|_{10}$) for paste with pH adjusted by adding various acids before gelatinization. $|\eta^*|_{10}$ of samples with $3.4 < \text{pH} < 6.0$ were greater than that of the control and $|\eta^*|_\omega$ of pastes, where pH is 6.0 and 3.4, were not different from that of the control, while $|\eta^*|_\omega$ of a sample with pH3.0 was less than that of the control. Comparison of $|\eta^*|_{10}$ in Fig. 5 with η_{10} in Fig. 1 shows that $|\eta^*|_\omega$ and $\eta_{\dot{\gamma}}$ of all samples did not obey the Cox-Merz rule; $\eta_{\dot{\gamma}} \neq |\eta^*|_\omega$ because starch pastes contains swollen granules and glucose chains. $|\eta^*|_{10}$ was greater than η_{10} on samples with $3.6 \leq \text{pH} \leq 5.3$. The cornstarch pastes with this pH range behaved as a weak gel. On the other hand, the cornstarch pastes, where pH is above 5.3 and below 3.6, behaved like a liquid.

From the results mentioned above, adding much acid leads to a decrease in the viscosity for starch pastes because the hydrolysis of many glucose chains occurs. When acids are added to the cornstarch pastes after heating and cooling, i.e., after gelatinization, the hydrolysis of glucose chains can be prevented. Therefore, higher viscosity for starch pastes should be obtained.

3.5. The effects of adding acids after gelatinization

Fig. 6 shows $\eta_{\dot{\gamma}}$ of 3.0 wt% cornstarch pastes with pH adjusted by lactic acid after gelatinization. All samples showed typical shear-thinning behaviors. There were no differences between the control sample and samples with pH6.0. Moreover, decreases in the viscosity could not be observed even at lower pH (pH = 3.0). Many starch granules could be seen in the microscopic photographs for the sample adjusted to pH3.5 and 3.0 after gelatinization, compared with those adjusted to pH before gelatinization (bottoms in Fig. 2). The values of $\eta_{\dot{\gamma}}$ for all samples were greater than that of the control except for the samples with pH6.0.

The same tendencies were observed on dynamic oscillatory measurements. No decrease in $|\eta^*|_{\omega}$ was observed when the pH was adjusted by acetic acid after gelatinization (Fig. 7). Similar tendencies were observed for all samples with pH adjusted by adding various acids (data not shown). $|\eta^*|_{\omega}$ was greater than $\eta_{\dot{\gamma}}$ on samples with pH < 6.0. The cornstarch pastes with this pH range behave as weak gels. This behavior indicates that there are many entanglements of glucose chains accompanied with starch granules in this pH range.

4. Conclusions

When sour seasonings (acids) were added before gelatinization, the viscosity of cornstarch pastes increased by adjusting the pH between 5.5 and 3.6, while the viscosity of pastes with pH below 3.5 decreased further than that of the control (pH = 6.3) in the steady shear and dynamic oscillatory measurements. The viscosity of starch pastes depended on the pH since similar tendencies were observed by adding various acids.

It is obvious from intrinsic viscosity measurements that the hydrolysis of glucose chains occurs by adding acid before gelatinization. However, increases in the viscosity could be seen by adding small amounts of acids ($3.5 < \text{pH} < 5.5$), because many glucose chains leached out from starch granules, inducing entanglements of glucose chains. On the other hand, adding large amounts of acids ($\text{pH} \leq 3.5$) leads to the collapse of starch granules and thus, decrease in the viscosity was observed.

No decrease in the viscoelasticity was noted for cornstarch pastes by adding acids after gelatinization. The hydrolysis of glucose chains could be prevented.

Acknowledgments

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Figure captions

Fig. 1. pH dependence of shear viscosity at the shear rates of 10 s^{-1} (η_{10}) for 3.0 wt% cornstarch pastes. The pH was adjusted by adding various acids before gelatinization. Measurements were made at 25.0 °C.

Fig. 2. Microscopic photographs of starch granules for 3.0 wt% cornstarch pastes. Pastes were diluted with distilled water and dyed with 10 mM iodine solution. The pH was adjusted by adding citric acid. Image size is $390\mu\text{m} \times 725\mu\text{m}$. Observations were made at ambient temperature.

Fig. 3. Intrinsic viscosity ($[\eta]$) of cornstarch at various pH in 90 vol% DMSO aqueous solution. The pH was adjusted by various acids before gelatinization. Measurements were made at 40.0 °C.

Fig. 4. Frequency (ω) dependence of A) G' and B) G'' for 3.0 wt% cornstarch paste. The pH was adjusted by adding tartaric acid before gelatinization. Measurements were made at 25.0 °C The strain for samples with pH6.3 (control), 6.0, 5.0, 4.0, and 3.0, was 30, 30, 3, 3, and 20 %, respectively.

Fig. 5. pH dependence of complex viscosity ($|\eta^*|_\omega$) of 3.0 wt% cornstarch pastes. The pH was adjusted by adding various acids before gelatinization. Measurements were made at 25.0 °C.

Fig. 6. Shear viscosity ($\eta_{\dot{\gamma}}$) of 3.0 wt% cornstarch pastes. The pH was adjusted by adding lactic acid after gelatinization. Measurements were made at 25.0 °C.

Fig. 7. Complex viscosity ($|\eta^*|_\omega$) of 3.0 wt% cornstarch pastes. The pH was adjusted by adding acetic acid after gelatinization. The strain for samples with pH6.3 (control), 6.0, 5.0, 4.0, and 3.0, was 30, 30, 3, 3, and 3%, respectively. Measurements were made at 25.0 °C.

Figures

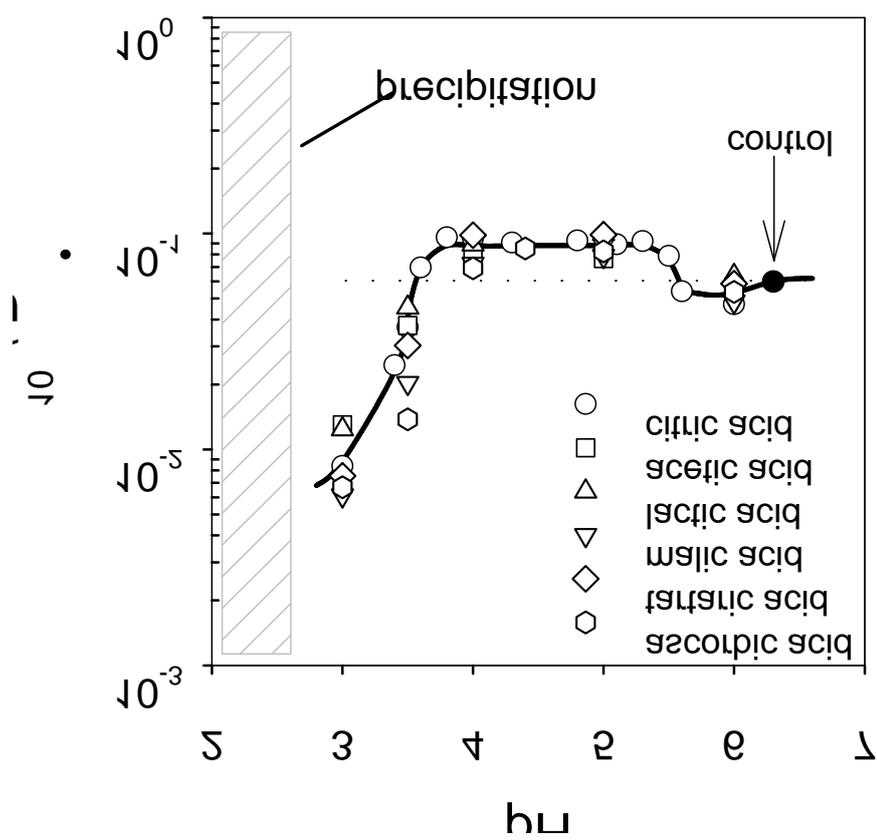


Fig. 1.

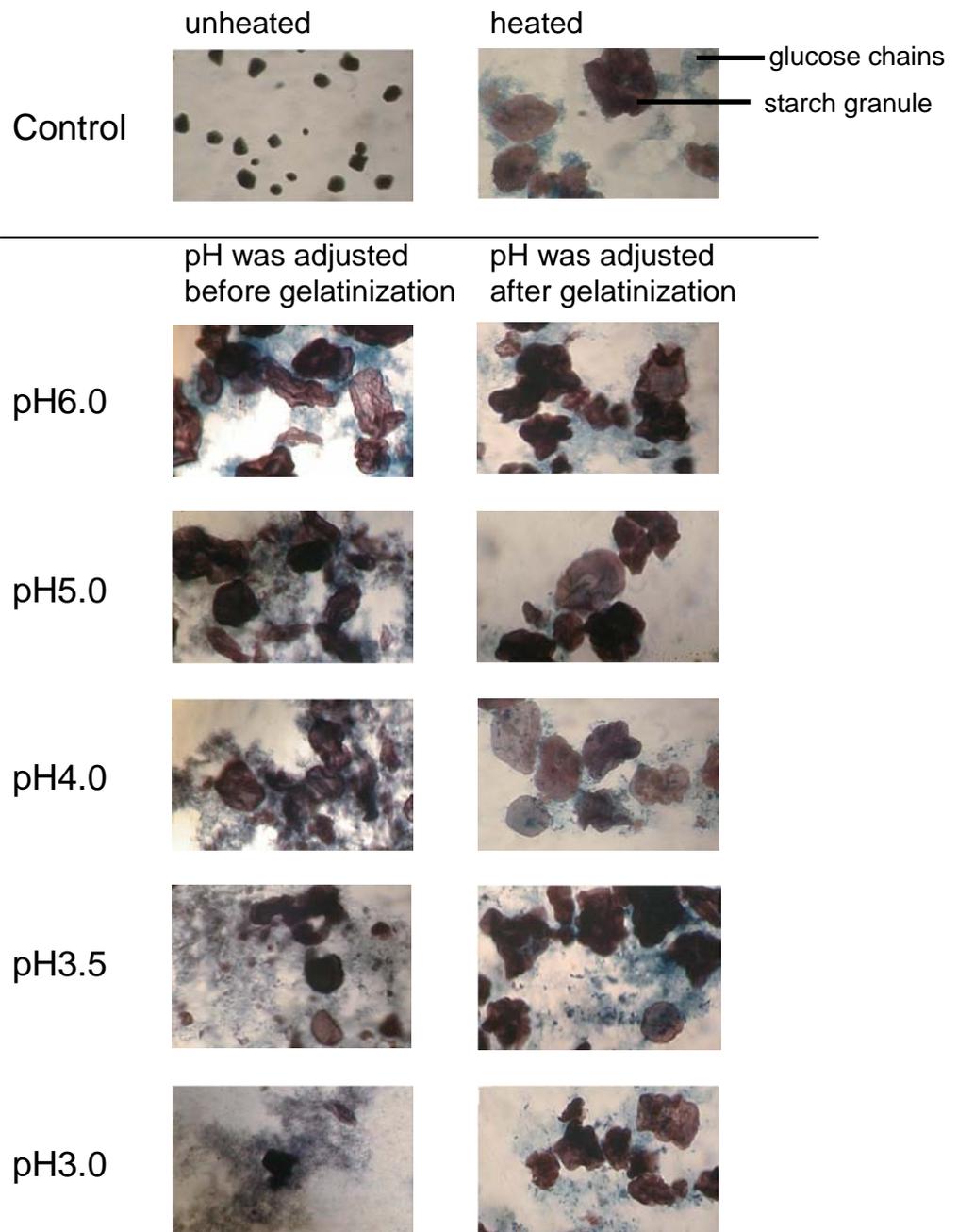


Fig. 2.

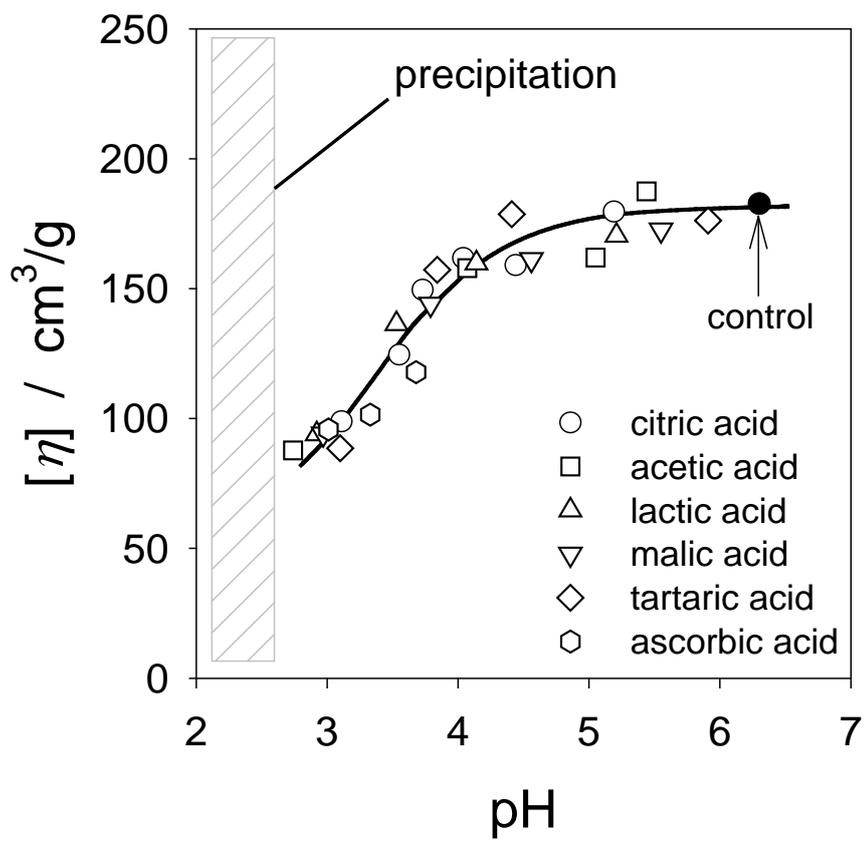


Fig. 3.

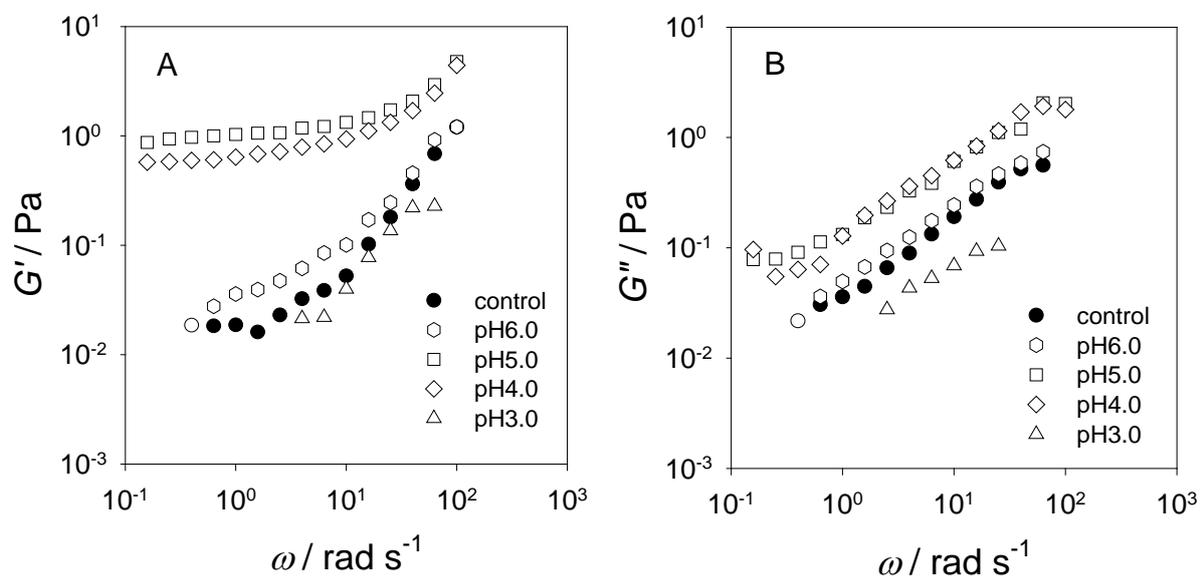


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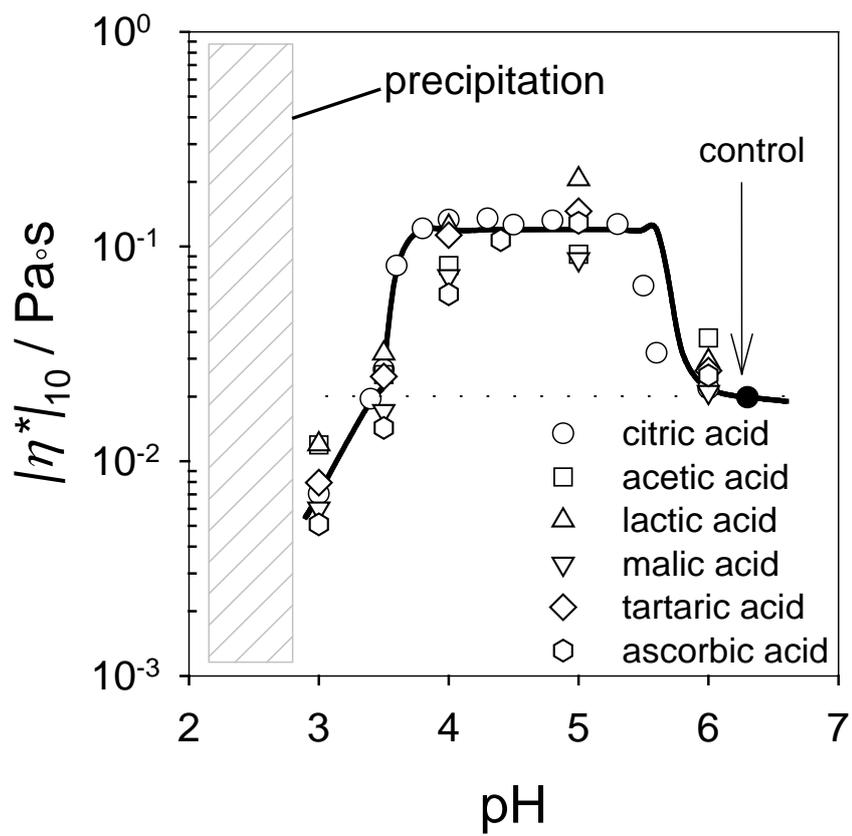


Fig. 5.

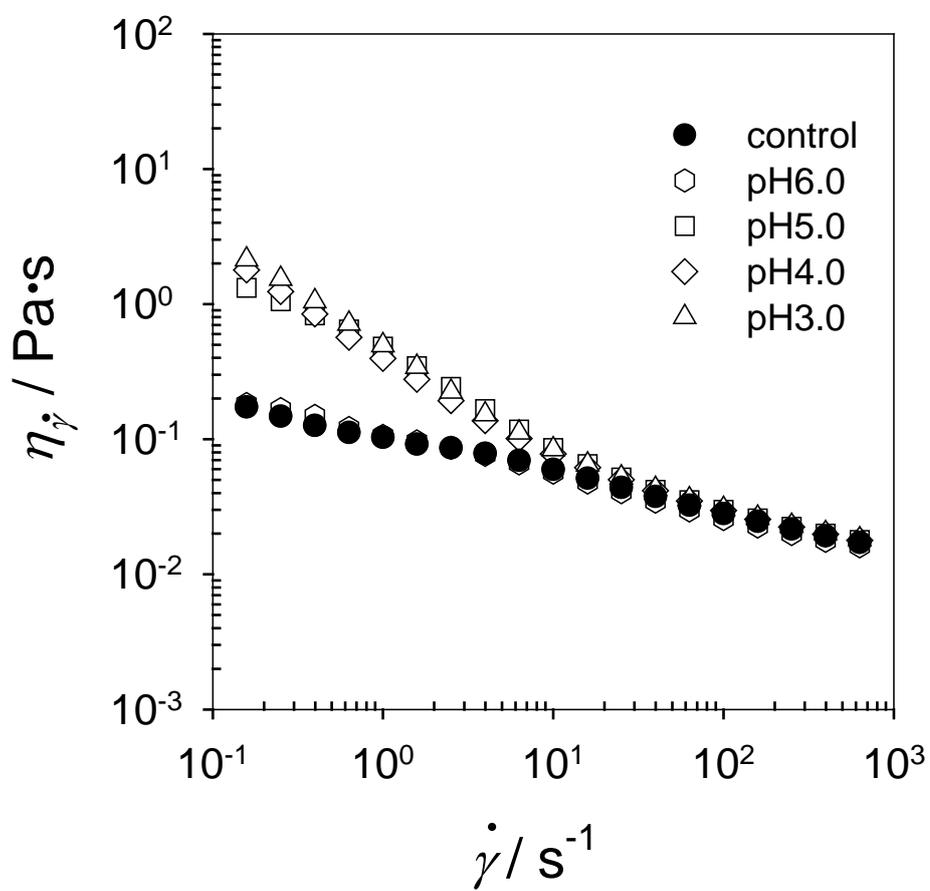


Fig 6.

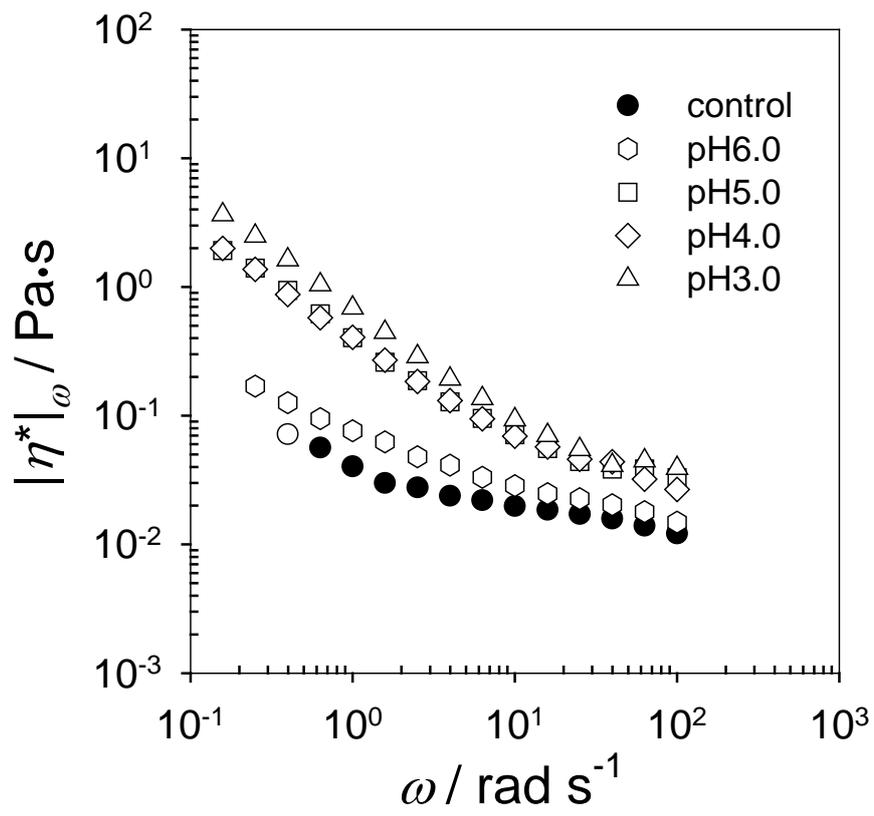


Fig. 7.