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Gel formation in mixtures of high amylopectin potato starch and potato starch

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Abstract

The effects of starch granules on the rheological behaviour of gels of native potato and high amylopectin potato (HAPP) starches have been studied with small deformation oscillatory rheometry. The influence of granule remnants on the rheological properties of samples treated at 90 °C was evident when compared with samples treated at 140 °C, where no granule remnants were found. The presence of amylose in native potato starch gave to stronger network formation since potato starch gave higher moduli values than HAPP, after both 90 and 140 °C treatments. In addition, amylose may have strengthened the network of HAPP because higher moduli values were obtained when native potato starch was added to the system. The moduli values of the mixtures also increased with increasing polysaccharide concentration in the system, which is due to an increment in the polysaccharide chain contacts and entanglements. Finally, it was found that a mixture of commercial amylose from potato starch and HAPP resulted in lower values of G' compared to native potato starch. This indicates that the source of amylose is important for the properties in a blend with native amylopectin.

Keywords: Amylopectin; Polysaccharide; Starch; High amylopectin potato starch; Potato starch

1. Introduction

Starch has been extensively studied because it is an inexpensive raw material in many industrial processes, where it is used mainly as a gelling, thickening, and forming agent. The most common form of starch used in the industry is as a gelatinised dispersion which is then mixed with other components in order to obtain a final product.

Starch samples, prepared in various ways and with different solvents, are commonly referred to as starch dispersions or solutions. However, a direct comparison of the sample characteristics, behaviour and properties is sometimes very difficult, if ever possible. This is because there is a lack of information regarding their actual physical state.

Several rheological studies on starch samples prepared in different ways are reported in the literature (Bagley & Christianson, 1982; Evans & Lips, 1992; Okechukwu & Rao, 1998; Visser, Suurs, Bruinenberg, Bleeker, & Jacobsen, 1997). The viscoelasticity and shear thinning flow behaviour of gelatinised starch dispersions are strongly influenced by the starch granule mass fraction (Evans & Haisman, 1979; Noel, Ring, & Whittam, 1993), granules size, size distribution (Eliasson, 1980; Evans & Lips, 1992; Okechukwu & Rao, 1998; Steeneken, 1989; Wong & Lelievre, 1981), shape, granule interactions, viscosity of the continuous phase, rate of deformation, and the formation of amylose-lipid complexes and their crystalline packing (Nelles, Dewar, van der Merwe, & Taylor, 2003).

Because there are many possibilities to influence this complex system, it is of uttermost importance to understand the role of each component in the starch system. The granule structure in the sample is of special importance, and should be taken into account before any measurement is performed. It has been claimed that remaining granule rests or remnants do affect the sample rheological properties (Genovese & Rao, 2003; Howling, 1980; Rao, Okechukwu, Da Silva, & Oliveira, 1997; Ring, 1985), however, it is still common practice to think and assume that a starch dispersion behaves like a starch solution.

The intention of the present work was to study the influence of starch granules on the rheological behaviour of

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gels of dispersed or dissolved high amylopectin potato starch (HAPP) and potato starch. The gelation of HAPP and potato starch alone, and their mixtures were investigated using small deformation oscillatory rheometry. Because a rich amylopectin source (HAPP), was used in the study, it was possible to separate the effect of particles (granules) from the influence of the amylose leaching.

2. Materials and methods

2.1. Materials

Amylopectin (HAPP; $\ll 1\%$ amylose) and potato starch (Prevalent-96 Native, 20.4% amylose) were provided by Lyckeby-Stärkelsen (Lyckeby, Sweden).

2.2. Methods

2.2.1. Rheological measurements

The rheological behaviour of potato starch samples was studied in potato starch suspensions (2, 3, 3.8, 4, 4.2, 6, 10, and 20%) prepared in water. HAPP:potato starch blends at 10 and 15% total polysaccharide concentration and at different HAPP:potato starch ratios (0:100; 20:80; 50:50; 80:20 and 100:0 HAPP:potato starch) were prepared with water. Samples were prepared following the previously established preparation procedure for HAPP to reduce the amount of granular remnants in the blend (Ortega-Ojeda, Larsson, & Eliasson, 2003b). Therefore, the suspensions were heated at 140 °C for 20 min, and then quenched for 4 s in a water bath at 60 °C, prior the rheological measurements.

HAPP–potato starch (2%, 100:0; 50:50; and 0:100 HAPP:potato starch) samples were prepared in water, heated at 90 °C for 20 min, and quenched for 4 s in a water bath at 60 °C, before the rheological measurements.

The samples were then transferred to the cone-plate geometry of a StressTech controlled stress rheometer (Reologica AB, Lund, Sweden), where the analysis was carried out at 10 °C. The storage and loss moduli (G' and G'') were measured as function of time (t) at 0.025 strain at a frequency (f) of 0.2 Hz. Decreasing the used frequency increases the measuring time, thus for practical reasons we selected 0.2 Hz. Furthermore, it should be taken into account that presenting the curing time processes at lower frequencies would yield different results. The frequency dependence of G' and G'' (n' and n'') were determined from the power law relationships of G' and G'' in the frequency sweeps: $G' = G'_0\omega^{n'}$ and $G'' = G''_0\omega^{n''}$, where G'_0 and G''_0 are the intercepts, $\omega = 2\pi f$ is the frequency in rad·s^{-1}. The use of n' and n'' is a convenient way to represent the liquid-to-solid transition of the system. For n', values close to 2 or 0 represent liquid or solid behaviours, respectively, and for n'', values close to 1 or 0 represent liquid or solid behaviours, respectively. After preparation and before the actual rheological measurements started, the HAPP-containing samples were left to rest in the rheometer for 6 h for relaxation of residual stresses. Then, the rheological tests were performed for another 8 h, consequently, 14 h are reported as total curing time for HAPP samples. Potato starch samples only needed a relaxation time of 1 min, thus only 8 h are reported as curing time for potato starch. All data presented are the average of at least three different runs and sample preparations, and the standard deviation, was maximum 30 mPa.

3. Results and discussion

3.1. Influence of granule remnants on potato starch and HAPP

Fig. 1a shows the curing process for 2% potato starch and 2% HAPP treated at 90 and 140 °C, respectively. The storage modulus (G') of the potato starch samples treated at 90 °C was 20 times larger than for the potato starch samples treated at 140 °C. The G' values for HAPP treated at 90 °C were 13 times larger than the samples treated at 140 °C. The moduli for potato starch were always larger than the moduli for HAPP treated at both temperatures. For both potato starch and HAPP samples treated at 90 °C, G' was always larger than G'', whilst G' was smaller than G'' for the potato starch and HAPP samples treated at 140 °C. Hence the phase angle (δ), Tan $\delta = G''/G'$ was higher at 140 than at 90 °C. In all cases the systems showed stability with curing time at 10 °C.

Fig. 1b shows the mechanical spectra for the samples shown in Fig. 1a. A clear difference between the temperature treatments can be noticed, since two different behaviours are shown in the mechanical spectra. Samples treated at 90 °C showed solid-like behaviour (G' larger than G''), and only a slight frequency dependence in the entire frequency range. The necessary relaxation time for the samples treated at 90 °C was of the order of at least 10^4 s. On the other hand, samples treated at 140 °C showed liquid-like behaviour and strong frequency dependence.

It should be noticed that the G' value for the 2% HAPP sample treated at 90 °C was equivalent to the value for 12% HAPP (Ortega-Ojeda, Larsson, & Eliasson, 2003a), and 6% native potato starch (Fig. 2), both treated at 140 °C. The granule remnants are amylopectin-enriched structures, and could be described as swollen hydrated polymer composites, that behave as filler particles reinforcing the continuous matrix of entangled molecules (Biliaderis, 1998; Liu & Lelièvre, 1992; Rao et al., 1997; Ring, 1987). Therefore, due to the absence of amylose in the HAPP system, the higher G' values when treated at 90 °C clearly indicate the presence of granule remnants in the system. The G' value for a 50:50 HAPP:potato starch mixture treated at 90 °C (results not shown) was equivalent to the G' for 14% HAPP and 8% potato starch treated at

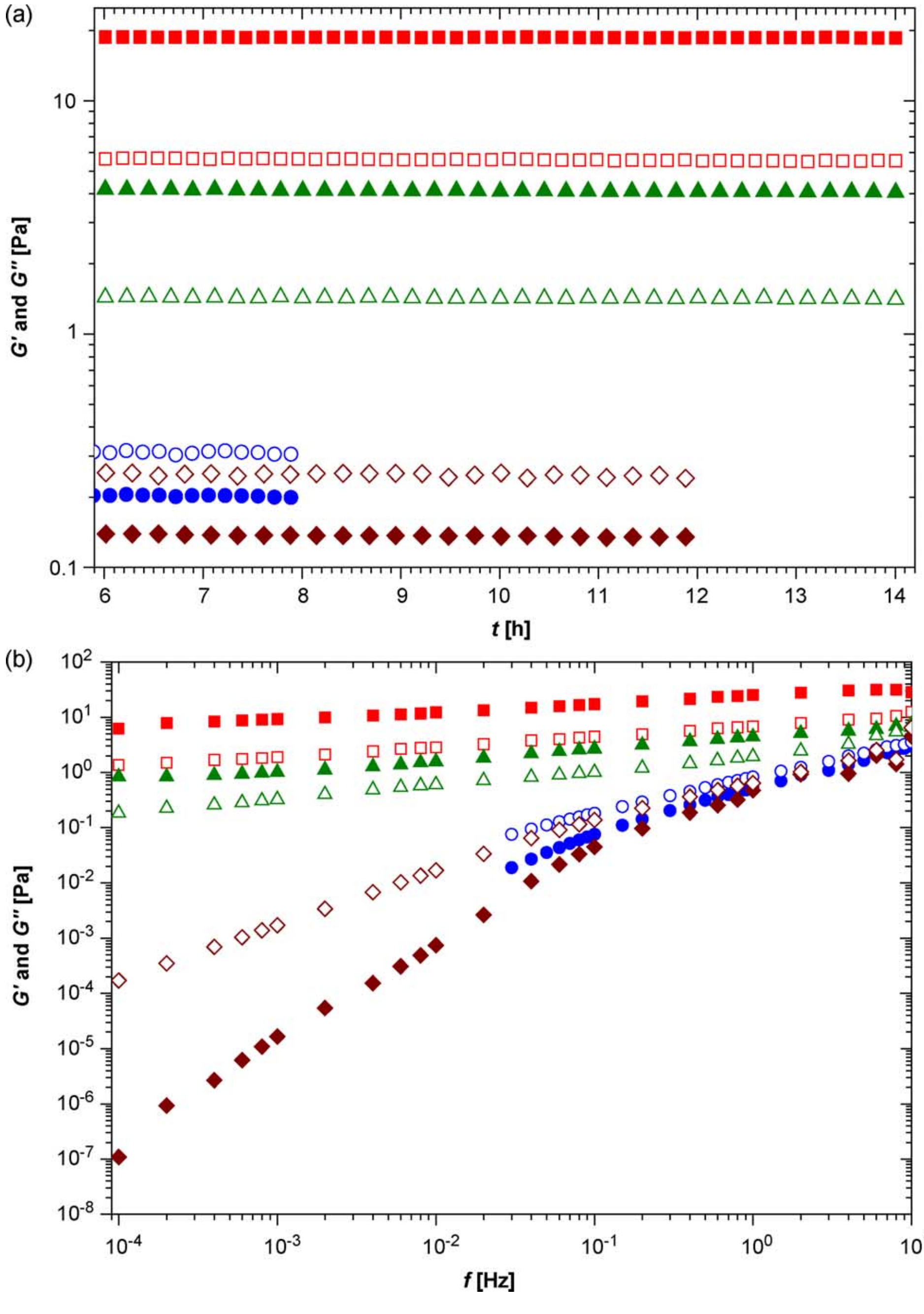

Fig. 1. (a) Storage modulus against time for 2% potato starch and HAPP samples treated at 90 or 140 °C, quenched to 60 °C and measured at 10 °C with 0.025 strain and at 0.2 Hz. (b) Mechanical spectra for the 2% potato starch and HAPP samples. (■, □), potato starch 90 °C; (●, ○), potato starch 140 °C; (▲, △), HAPP 90 °C; (◆, ◇), HAPP 140 °C. Closed symbols are G', and for the frequency sweep the open symbols are G''.

140 °C. Moreover, the profound effect of amylose and granule remnants on the rheological properties of the blend is indicated by the fact that only 2% potato starch treated at 90 °C (Fig. 1) was enough to increase G' to values comparable to those of ~25% HAPP (by extrapolation in Fig. 3) and ~13% potato starch, treated at 140 °C (Fig. 2).

3.2. Influence of amylose content on potato starch

Fig. 2a shows the curing process during 8 h for potato starch samples (2–20%) at 0.2 Hz, prepared at 140 °C. The value of G' increased with increasing concentration, and also with time during the first 20 min of curing. The time effect was stronger with increasing potato starch concentration; hence it was not possible to record at 10 and 20% potato starch. After the initial 30 min curing the systems were independent of time regardless of the concentration. In a previous report (Muhrbeck & Eliasson, 1991), a similar time dependence was mentioned.

Fig. 2b shows the mechanical spectra of the potato starch samples at 2–20% at the end of the measurements shown in

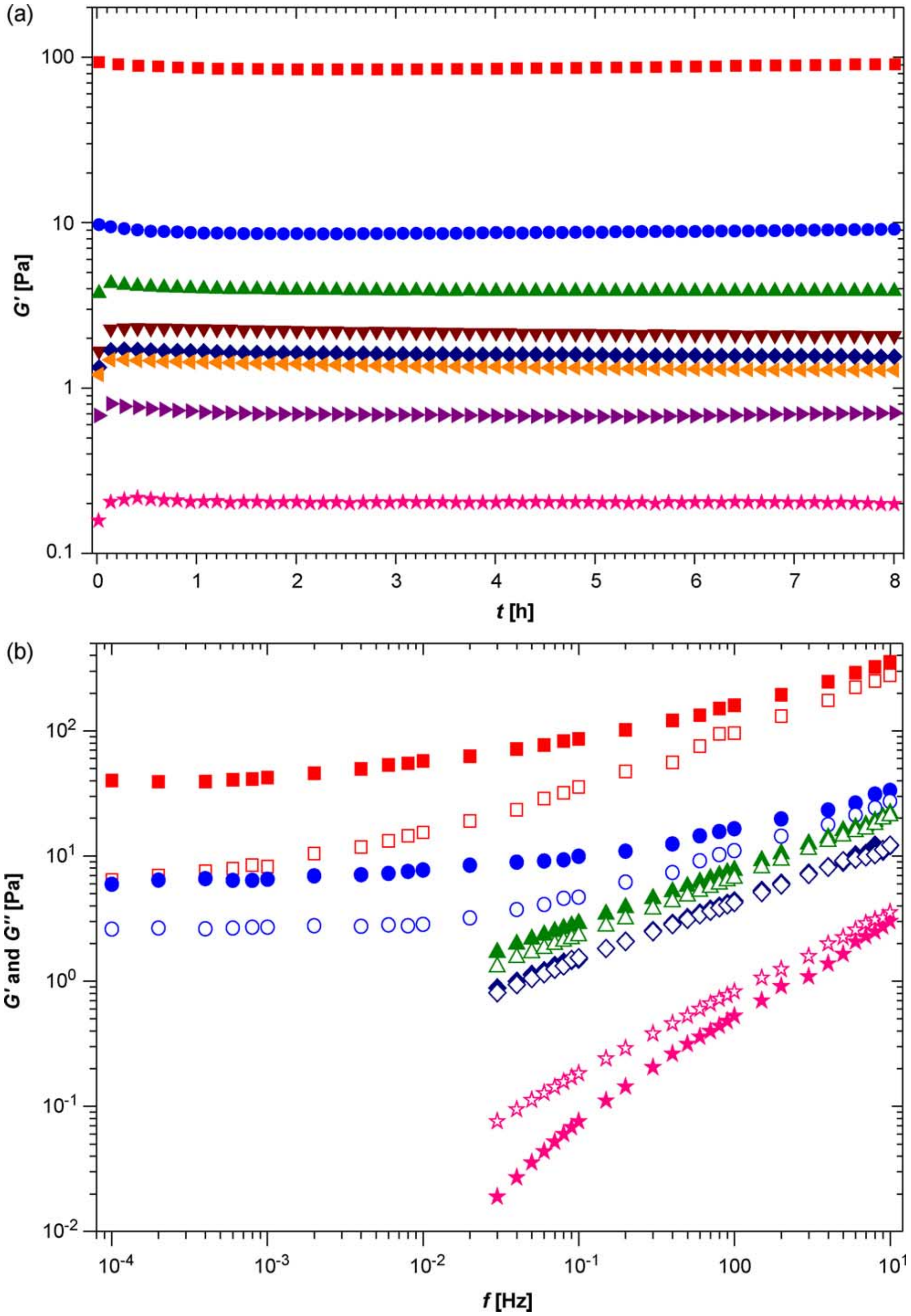

Fig. 2. (a) Storage modulus against time for potato starch samples without granule remnants, at different concentrations, after heating at 140 °C, quenching to 60 °C and measured at 10 °C with 0.025 strain and at 0.2 Hz. (b) Mechanical spectra for the potato starch samples. (■, □), 20%; (●, ○), 10%; (▲, △), 6%; (▼, ▽), 4.2%; (◆, ◇), 4%; (◄, ◁), 3.8%; (►, ▷), 3%; (★, ☆), 2%. Closed symbols are G', and for the frequency sweep the open symbols are G''.

Fig. 2a, i.e. after 8 h at 10 °C. Samples with $>4\%$ potato starch showed solid-like behaviour (G' larger than G''), which was not the case at concentrations lower than 4%. All samples showed frequency dependence, which decreased slightly with increasing potato starch concentration. The necessary relaxation time for the systems at concentrations $>10\%$ was long, of the order of at least 10^4 s.

In Fig. 3a the moduli after the total curing time for potato starch are compared with the corresponding values for HAPP, and commercial amylose (Ortega-Ojeda et al., 2003a) as a function of polysaccharide concentration (all samples prepared at 140 °C). At the same concentration, the values of G' for potato starch were larger than for HAPP. This agrees with a previous study (Visser, Suurs, Steeneken,

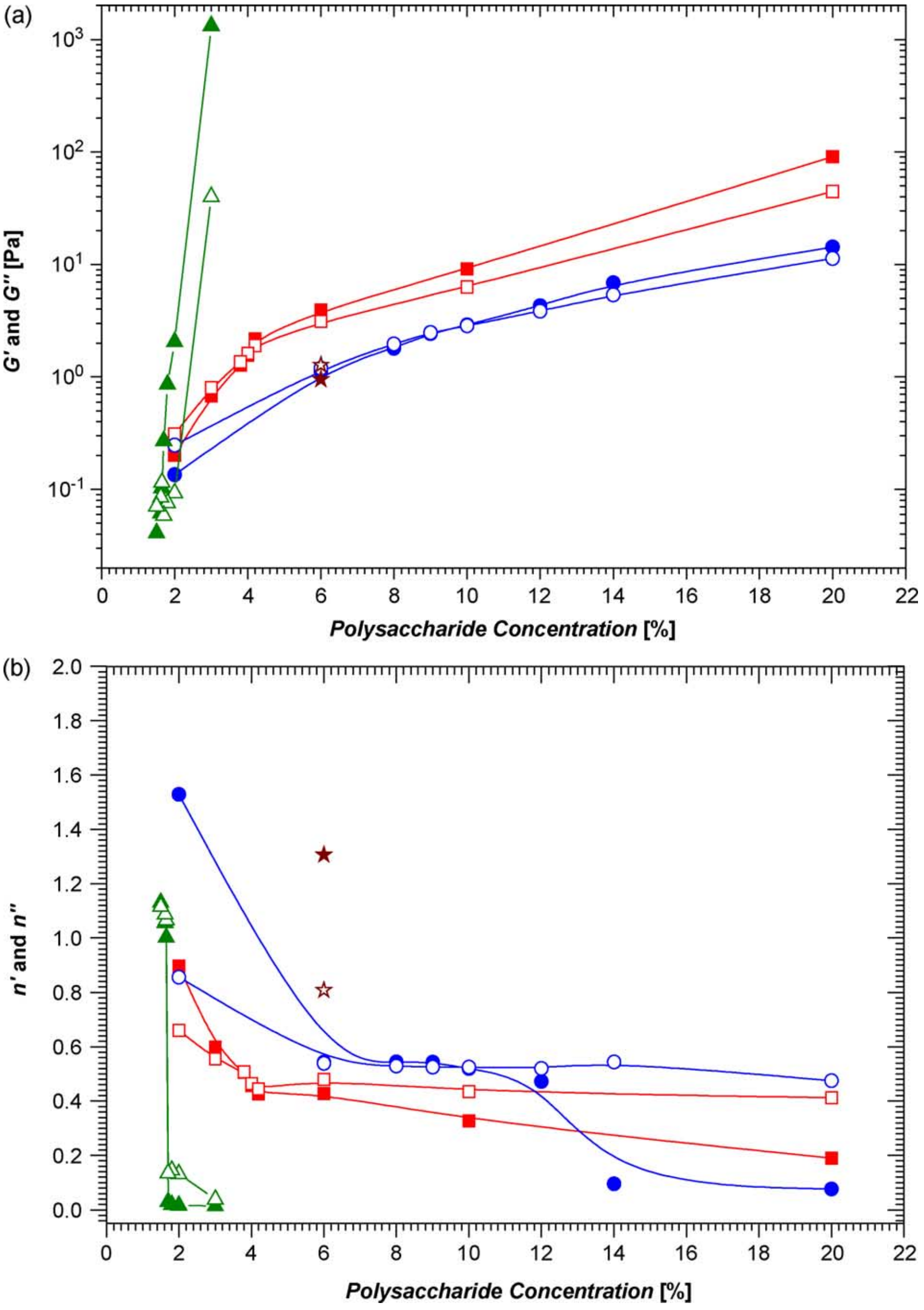

Fig. 3. (a) Comparison of the limiting moduli (G' and G'' after the curing time) against total polysaccharide concentration for potato starch, HAPP, amylose, and 6% HAPP:amylose samples. (b) Frequency dependence versus total polysaccharide concentration for the samples shown in (a). (■, □), potato starch; (●, ○), HAPP; (▲, △), amylose; and (★, ☆), 6% HAPP:amylose (80:20 HAPP:amylose ratio) (Ortega-Ojeda et al., 2003a). Closed symbols are G', and the opened symbols are G''.

& Jacobsen, 1997) which reports that the G' values for the amylose-free potato starch prepared at 90 °C were lower than those of amylose containing potato starch. However at the same concentration, the moduli values for potato starch and HAPP were not as high as for amylose. A lower minimum concentration (1.6%) was required for amylose to observe $G' > G''$ (Ortega-Ojeda et al., 2003a). On the other hand, potato starch and HAPP showed $G' > G''$ only when concentration was approximately higher than 4 and 9%, respectively. For example, the value of G' for ~2% amylose, was equivalent to the value of G' for ~4.2% potato starch and ~9% HAPP (Fig. 3a). At polysaccharide concentrations above where $G' \approx G''$, the G'/G'' ratio was slightly larger for potato starch than for HAPP. This as well indicates the formation of a denser network in case of potato starch. The values of the storage modulus at the highest potato starch concentrations were almost seven times larger than the values for HAPP at the same concentrations.

Fig. 3b shows the frequency dependence for G' and G'' (n' and n'', respectively) as a function of polysaccharide concentration for potato starch, HAPP, and amylose. For potato starch the values of n' decreased in two steps, i.e. abruptly from ~ 0.9 to ~ 0.4 when the concentration increased from 2 to 4% potato starch, and then up to 20% polysaccharide concentration the decrease in n' was only moderate. Thus, the liquid-to-solid transition did not show a strong concentration dependence between 4 and 20%. The corresponding transition for HAPP resembled that of potato starch, but took place in three different steps (Ortega-Ojeda et al., 2003b). For HAPP, the frequency dependence of G' decreased strongly from ~ 1.6 to a constant value of about 0.5, for HAPP concentrations increasing from 2 to 6%. Between 6 and 12% HAPP, n' is approximately equal to n''. At higher concentrations than 12% HAPP, n' decreased even more, approaching zero, which implies a solid-like behaviour. For both potato starch and HAPP, n'' decreased slowly, reaching a more or less constant value with increasing concentration (Ortega-Ojeda et al., 2003a). The stronger transition in n' and n'' (from liquid to solid) at a lower concentration for potato starch compared with HAPP, could be attributed to the presence of amylose in potato starch. However, the liquid-to-solid transition observed for the potato starch sample was yet less drastic than for pure amylose. Furthermore, the presence of more amylose in potato starch at 20% does not account for the value of n', which was higher than for HAPP at the same concentration. Interestingly, the values of n' and n'' for the samples containing 2% total polysaccharide, treated at 90 °C, were almost as high as the values for the samples containing 20% polysaccharide, treated at 140 °C. This shows the impact of the presence of amylose and granule remnants on a sample.

Now when the viscoelastic properties of potato amylose, potato amylopectin (i.e. HAPP), their mixtures, and potato starch are available, we can evaluate if the behaviour of potato starch is achieved in a mixture of the individual components. This was checked for samples with 6% total polysaccharide concentration, and included in Fig. 3 are the moduli for a 6% mixtures of HAPP and commercial amylose (Ortega-Ojeda et al., 2003a) with the same composition as native potato starch, i.e. 20% amylose is more or less equivalent to the moduli of 6% HAPP sample. In other words, the viscoelastic behaviour of native potato starch could not be reconstituted by a blend of HAPP and commercial amylose from potato starch. It is clear that the native amylose present in the potato starch samples ($\sim$20%) greatly influenced the rheological behaviour, reinforcing the growing network and thus the augment of the storage modulus. This effect was probably further accentuated due to an increase in chain contacts and entanglements that were induced when the potato starch concentration increased. The lack of amylose in HAPP led to a smaller increase in the moduli although the HAPP concentration was increased. Probably the poor interpenetration, if any, of the outer branches of amylopectin molecules was not enough to produce the same effect on the system as the amylose chains.

3.3. Influence of amylose content in HAPP–potato starch mixtures

Fig. 4a shows the values of G' versus time for HAPP–potato starch mixtures at a total polymer concentration of 10% and different HAPP:potato starch ratios, at 0.2 Hz during 8 h curing at 10 °C. The values of G' increased with increasing potato starch concentration. The storage modulus showed slightly lower stability with time for the samples containing more potato starch (0:100 to 50:50 HAPP:potato starch), i.e. when the content of HAPP decreased.

Fig. 4b shows the mechanical spectra for HAPP–potato starch mixtures at 10% polysaccharide concentration, after 8 h curing. All samples showed frequency dependence, which decreased slightly with increasing concentration of potato starch. Nevertheless, all mixtures presented solid-like behaviour. Besides, the relaxation time for all the blends was of the order of at least 10^4 s.

Fig. 5a shows the values of G' versus time at 0.2 Hz during 8 h curing at 10 °C for HAPP–potato starch mixtures at a total polymer concentration of 15% and different HAPP:potato starch ratios. G' increased with increasing potato starch concentration but not in linear fashion. As for the 10% HAPP–potato starch mixtures, G' for the 15% HAPP:potato starch blends containing 50:50, 20:80 and 0:100 HAPP:potato starch, the storage modulus increased slightly during the curing time. Furthermore, the difference in G' found between the samples containing 20:80 and 0:100 HAPP:potato for 10% total concentration, has shifted towards HAPP at 15%, i.e. now is between 50:50 and 20:80 HAPP:potato.

Fig. 5b shows the mechanical spectra for HAPP–potato starch mixtures at 15% polysaccharide concentration, after 8 h curing. The moduli showed a slight frequency dependence, which decreased with increasing potato starch concentration. All mixtures showed solid-like behaviour ($G' > G''$), and less frequency dependence than the 10% HAPP–potato starch samples The relaxation time for the system was of the order of at least 10^4 s.

Fig. 6a shows the moduli of the HAPP:potato starch mixtures as a function of total amylose concentration in the sample at 0.2 Hz after 8 h of curing time at 10 °C. As the importance of the amylose source was pointed out above (Fig. 3), a 10% HAPP:amylose mixture (Ortega-Ojeda et al., 2003a) is included in Fig. 6 to compare the influence of native and commercial amylose. The values of G' and G'' increased for the HAPP–potato starch blends, with increasing total polysaccharide and amylose concentrations. The former is related to an increment in polymer contacts and entanglements between amylose and

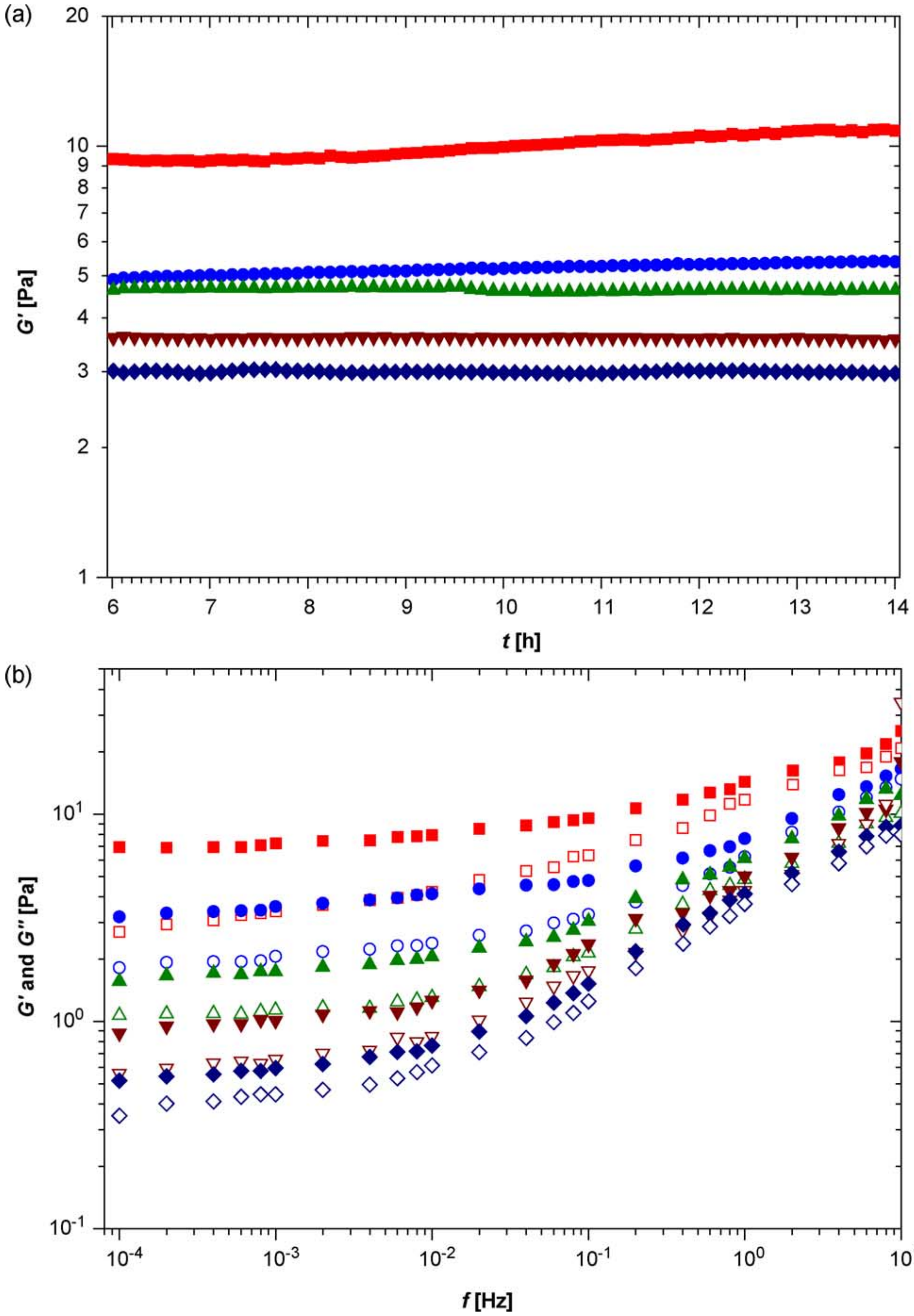

Fig. 4. (a) Storage modulus against time for 10% HAPP:potato starch solutions with different HAPP:potato starch ratio, after heating at 140 °C, quenching to 60 °C and measured at 10 °C with 0.025 strain and at 0.2 Hz. (b) Mechanical spectra for 10% HAPP:potato starch samples. HAPP:potato starch ratio: (■, □), 0:100; (●, ○), 20:80; (▲, △), 50:50; (▼, ▽), 80:20; (◆, ◇), 100:0. Closed symbols are G', and for the frequency sweep the opened symbols are G''.

amylopectin. The latter may be explained by the fact that amylose itself forms a strong network, which in turn favours the formation of the amylopectin network. This is because when amylose is not present in the system or present only in small amounts, a strong network is not favoured since amylopectin alone does not form a strong network. The increasing values of the moduli with increasing amylose concentration seemed to be more or less linear, regardless of the total polysaccharide concentration.

Moreover, in Fig. 6a we can see that for a sample with a total polysaccharide concentration of 10%, only ~1% native amylose from potato starch (50:50 HAPP:potato starch) was needed to get the same G' value as was obtained with ~3.6% commercial amylose (~36:67 HAPP:Am). This may be explained by differences in molecular

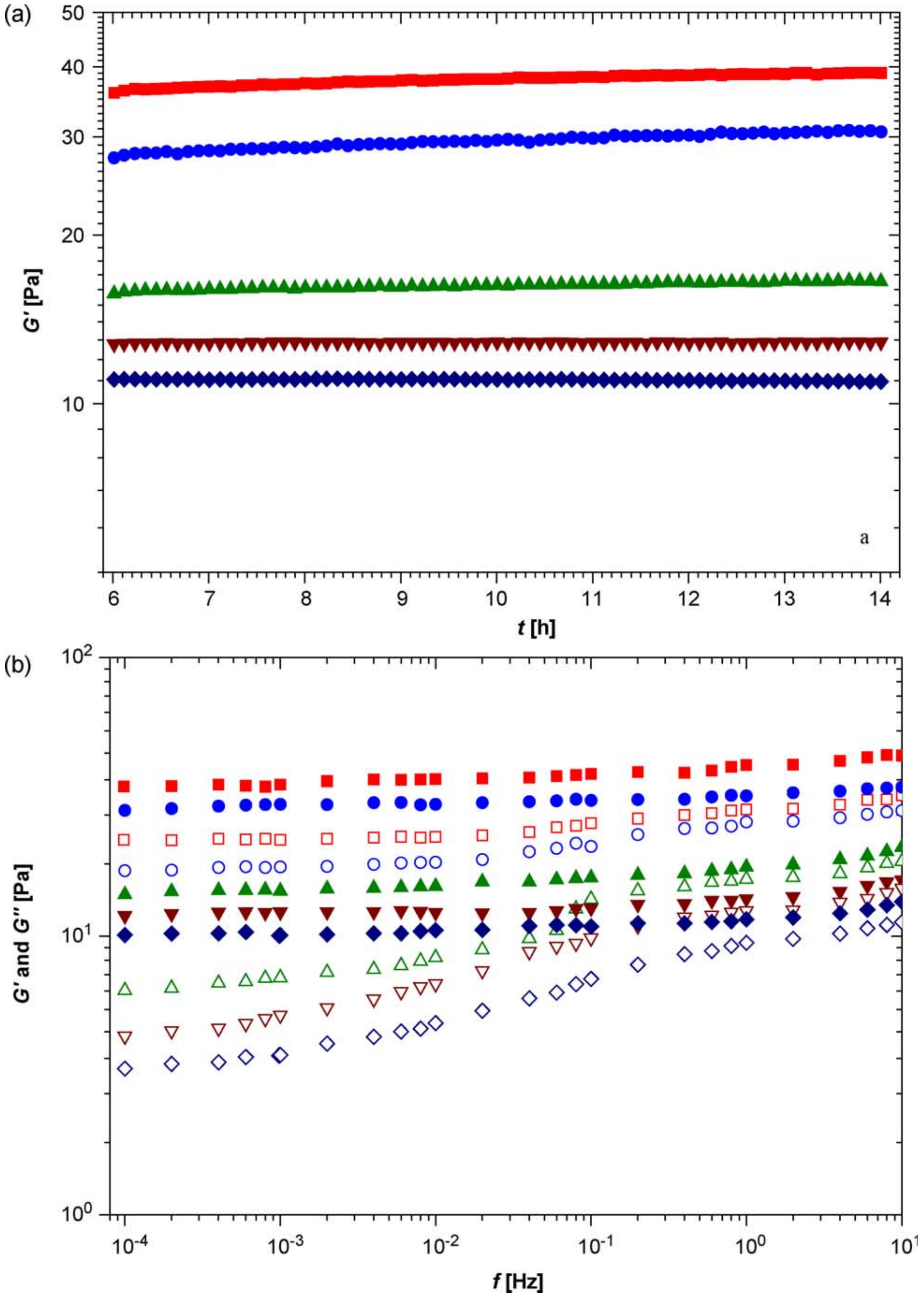

Fig. 5. (a) Storage modulus against time for 15% HAPP:potato starch solutions with different HAPP:potato starch ratio, after heating at 140 °C, quenching to 60 °C and measured at 10 °C with 0.025 strain and at 0.2 Hz. (b) Mechanical spectra for 15% HAPP:potato starch samples. HAPP:potato starch ratio: (■, □), 0:100; (●, ○), 20:80; (▲, △), 50:50; (▼, ▽), 80:20; (◆, ◇), 100:0. Closed symbols are G', and for the frequency sweep the opened symbols are G''.

composition and structure between the native amylose and the commercial one, where the latter has experienced extraction and purification.

Fig. 6b shows the frequency dependence for G' and G'' (n' and n'', respectively) as a function of total amylose in the sample for the mixtures shown in Fig. 6a. For all mixtures, n' decreased gradually with increasing total polysaccharide and amylose concentrations. This shows a slow transition from a weak network where only amylopectin is present to a stronger one, with amylose-amylopectin interactions. However the 2% HAPP:potato starch mixture, showed only liquid behaviour ($n' > n''$) in the concentration region investigated. In addition, the 10% HAPP:amylose mixture presented a much more concentration dependent transition from liquid to solid behaviour. However, much more amylose was needed in comparison with the 10% HAPP:potato starch blend.

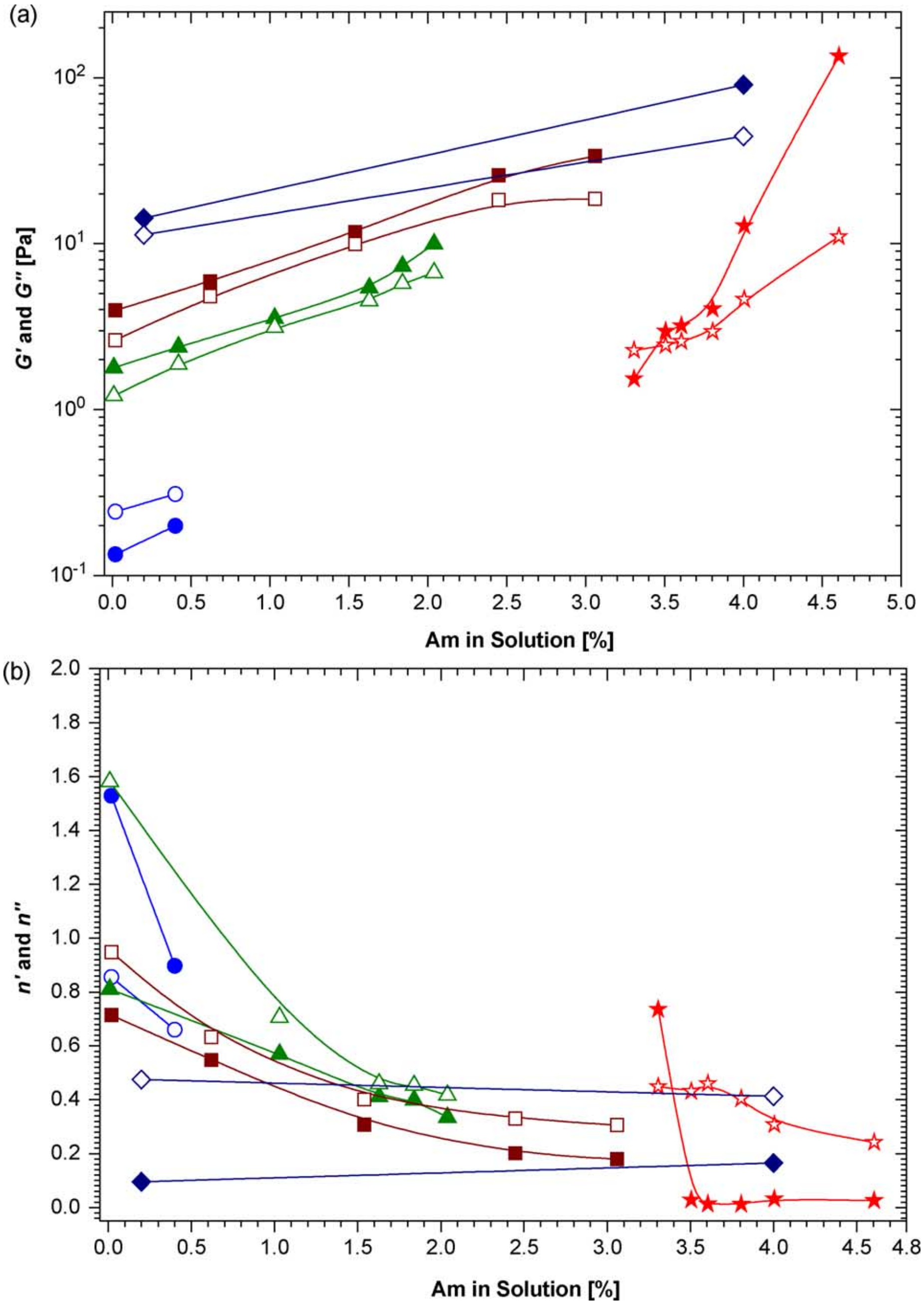

Fig. 6. (a) Comparison of the limiting moduli against total amylose in HAPP:potato starch (2–20 total concentration) and 10% HAPP:amylose samples (Ortega-Ojeda et al., 2003a), at different ratios, and treated at 140 °C. (b) Frequency dependence versus total amylose in solution for the samples shown in (a). (★, ☆), 10% HAPP:amylose; (●, ○), 2%; (▲, △), 10%; (■, □), 15%; and (◆, ◇), 20% HAPP:potato starch. Closed symbols are G', and the opened symbols are G''.

4. Conclusion

The influence of starch granules on the rheological behaviour of gels of HAPP and native potato starch was studied by means of small deformation oscillatory rheometry. It was clearly found that the presence of granule remnants in samples treated at 90 °C, does strongly influence the rheological properties of a starch system, in comparison with samples treated at 140 °C, where no granule remnants are detected.

A difference in the rheological behaviour of native potato starch and HAPP was noticed, since potato starch gave higher moduli values than HAPP. This should be related to the presence of amylose in native potato starch, which led to stronger network formation. When native potato starch was added to HAPP, higher moduli values were obtained due to the presence of amylose in the blends, which strengthen the network.

Furthermore, besides the presence of amylose in the sample, increasing polysaccharide concentration in the system also increased the moduli values of the mixtures. This would be expected since it increases the polysaccharide chain contacts and entanglements.

Finally, it was clearly shown that commercial amylose does not behave as native amylose in a blend with native amylopectin. A mixture of commercial amylose from potato starch and HAPP did not give the viscoelastic behaviour of native potato starch.

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