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Stress relaxation in synergistically associated polysaccharides: Galactomananns and a non-pectic polysaccharide fraction from yellow mustard mucilage

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ABSTRACT

In the current study, stress relaxation test and dynamic temperature sweep test were performed to study a non-pectic polysaccharide (NPP) fraction from yellow mustard mucilage and its synergistic interactions with galactomannans (GMs). All solutions were prepared at the total polysaccharide concentration of 1% (w/v). NPP and GM were mixed at the blending ratio of 7/3. Prior to measuring the behaviour of the mixed solutions, the effects of temperature and pH on 1% NPP solution were studied. The results showed that both acidic condition and increased temperature led to stronger NPP gels due to hydrophobic interactions along the NPP backbones. When NPP was mixed with either fenugreek gum (FG) or locust bean gum (LBG) at pH 8, synergistic interactions were observed at all examined temperatures. The relaxation modulus ($G(t)$) increased at higher temperatures. The difference in relaxation curves between the two mixed solutions, NPP/FG and NPP/LBG, at increased temperatures further confirmed the difference in their synergy mechanism: the breakdown of "hyperentanglements" in NPP/FG solution started at a lower temperature (50 °C) while the breakdown of "junction zones" via hydrogen bonding in NPP/LBG solution occurred at a higher temperature (70 °C). This paper provides further evidence for our previously proposed mechanisms of the synergistic interactions in NPP/FG and NPP/LBG mixed systems.

1. Introduction

A novel non-pectic polysaccharide (NPP) fraction, a β-1,4 linked glucan, was isolated from water soluble yellow mustard mucilage. The rheological properties of this fraction have been characterized as exhibiting weak gel structure at low concentrations (as low as 0.1%, w/v), stable at wide temperature range from 5 to 90 °C, as well as acid resistant (Wu, Cui, Eskin, & Goff, 2009a). It was also found that NPP could synergistically associate with different types of galactomannans (GMs) to form stronger gels (Wu, Cui, Eskin, & Goff, 2009b). The unique properties of this fraction invoked strong interest in further investigation and application of this biomaterial in different areas such as pharmaceutical, cosmetic and food products (Cui, Eskin, Wu, & Ding, 2006). An intensive study on structure formation and deformation mechanism of NPP under various conditions was motivated. In our previous studies, rheological properties of NPP and NPP/GM mixed solutions were investigated by dynamic experiments. However, dynamic measurements can only provide rheological properties at short times (high frequencies) while stress relaxation experiments could be more appropriate to test the long time regime (Roth, Vega, Vallés, & Villar, 2004). The dynamic experiments differ from transient because a sinusoidal stress or strain is applied instead of a constant stress or strain. The typical transient tests can be represented by creep and stress relaxation experiments. In creep tests, a constant stress is applied to a sample and the corresponding strain is measured as a function of time. In stress relaxation tests, a constant strain is applied. The stress required to maintain the deformation is measured as a function of time (Del Nobile, Chillo, Mentana, & Baiano, 2007). When a stress relaxation test is performed, different behaviour can be observed: ideal elastic materials do not relax whereas ideal viscous materials instantaneously show a relation to time. Viscoelastic solids gradually relax and reach an equilibrium stress greater than 0, whereas for viscous fluids, the residual stress vanishes to 0 (Steffe, 1996). To evaluate the relaxation curve, the applied stress is separated into two components, a relaxing stress component and a non-relaxing stress component. The relaxing component represents the viscous property and the non-relaxing component represents the elastic property. In order to discriminate the stress relaxation behaviours of different soft solid noodles, Bellido and Hatcher (2009) used the shape and residual value of the relaxation curve as the two essential features to analyze the stress relaxation behaviour. According to Wortmann and Schulz (1994, 1996), a wide variety of materials show a universal shape on

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the relaxation curve. Mitchell (1980) proposed mechanical models consisting of spring (representing the elastic component) and dashpot (representing viscous fluid) elements which are often used to explain rheological properties of hydrogels. He suggested that time dependent mechanical behaviour in stress relaxation was due to entanglement coupling of large polymer chains in a covalently crosslinked gel, and the breaking of "junction zones" in noncovalent crosslinked gels (hydrogen or ionic bonds). Nussinovitch, Normand, and Peleg (1990), Peleg and Pollak (1982), and Tang, Tung, and Zeng (1998), on the other hand, attributed the time dependent characteristics of gels to the release of hydraulic pressure induced within gel samples during loading. In order to describe the relaxation curves, Le, Lüpke, Pham, and Radusch (2003) used three parameters: the relaxation time, the relaxing stress component, and the non-relaxing stress component.

In the present study, the mechanism of structure formation/deformation of NPP in response to two factors, temperature and pH values, was evaluated by examining the temperature sweep curves and stress relaxation behaviour. Also, the mechanisms of synergistic interactions between NPP and GMs were further studied by investigating the stress relaxation behaviour of the mixed solutions at different temperatures. As indicated in our previous studies (Wu et al., 2009b), fenugreek gum (FG), a fully substituted galactomannan with a mannose to galactose ratio of 1:1, can form "hyperentanglements", which also penetrate in the NPP network thus enhancing the gel strength; locust bean gum (LBG), a galactomannan with a mannose to galactose ratio of 4:1, can form "junction zones" with NPP molecules and thus improve the gel strength. Two types of galactomannans, FG and LBG, were used in the mixed solutions to represent the two postulated synergistic mechanisms with NPP. The shape of the relaxation curve and the two stress components, relaxing stress component and non-relaxing stress component at the corresponding relaxation time, were adopted to interpret the stress relaxation data.

2. Experimental

2.1. Materials and sample preparation

The non-pectic polysaccharide (NPP) fraction was isolated as described by Wu et al. (2009a). NPP, FG and LBG solutions were prepared at concentrations of 1% (w/w). The samples were weighed and dispersed into 50 mM phosphate buffer at pH 8 and stirred at room temperature for 30 min followed by heating at 80 °C for 1 h, then cooled down to room temperature with constant stirring. Four phosphate buffers were prepared to represent different pH conditions (2, 4, 8 and 10) for testing 1% NPP solutions. NPP solution and GM solutions (at pH 8) were mixed at the blending ratio of 7:3 (w/w) and stirred at room temperature for 30 min.

2.2. Rheological measurements

The rheological measurements were performed on a strain controlled ARES Rheometer (TA Instruments, New Castle, DE, U.S.A.), on a parallel-plate geometry (50 mm) at gap size of 0.5 mm. Temperature sweeps were performed using oscillatory measurements over the temperature range from 5 to 80 °C with the frequency set at 1 Hz. Stress relaxation tests were conducted at strain of 0.7% for 3 min. Each test was repeated three times and the means were represented as the final results.

3. Results and discussion

3.1. Temperature–pH effect on 1% NPP solutions

The relaxations curves of 1% NPP solutions at four pH conditions at 5 °C are presented in Fig. 1. Although the pattern of curves was similar at all conditions, the modulus ($G(t)$) at lower pH conditions was always higher. These results are in agreement with our previous results from a dynamic study which indicated that a stronger gel could be obtained at stronger acidic conditions (Wu et al., 2009a).

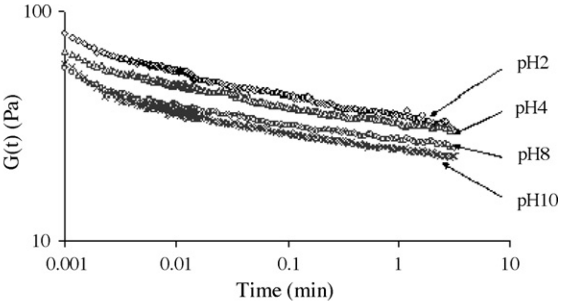

Fig. 1. Stress relaxation curves of 1% NPP solution at different pH conditions (NPP: non-pectic polysaccharide from yellow mustard mucilage).

Temperature sweeps at different pH conditions are given in Fig. 2. The temperature examined ranged from 5 to 80 °C. At pH 2, the shape of the sweep curve was different from that at the other three pH conditions by showing an obvious decrease of G' from 5 to 15 °C, a steady region from 15 to 40 °C, and a much steeper increase from 40 to 80 °C. At the other three pH conditions, the sweep curves showed a similar pattern with a smaller decrease of G' from 5 to 15 °C, a steady region from 15 to 40 °C and a smaller increase of G' from 40 to 80 °C. The G' at pH 2 was the highest throughout the temperature range studied, followed by pH 4. The temperature sweep curves for pH 8 and 10 overlapped for almost the entire temperature range. The results are consistent with that from the relaxation curves with the conclusion that NPP can form stronger gels at stronger acidic environments. The extraordinary jump of G' values, especially at the last stage of temperature sweep at pH 2, may be due to the response of the NPP molecules to the specific pH condition. The conformation of NPP molecules under these conditions might favour more hydrophobic interactions.

The effect of temperature on the NPP networks was investigated by relaxation tests at different temperatures: 5, 10, 15, 20, 30, 40, 50, 60 and 70 °C. The results are shown in Fig. 3. The relaxation curves obtained at 5 °C and 30 °C overlapped, indicating similar amount of structure in the solution. At 10, 15 and 20 °C, although the initial $G(t)$ values were close to that at 5 °C, the curves diverged at longer times. Temperature is an important factor determining the molecular mobility of the material. An increase in the temperature accelerates thermally activated processes and gives faster relaxation (Le et al., 2003). The increase of temperature from 5 to 15 °C may improve the mobility of the molecules; meanwhile, the enhanced mobility of the molecules can facilitate the hydropho-

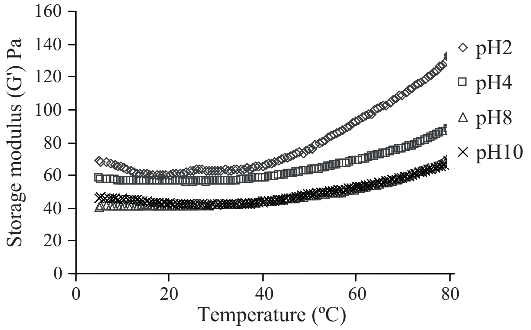

Fig. 2. Storage modulus of 1% NPP solutions as a function of temperature at different pH conditions (NPP: non-pectic polysaccharide from yellow mustard mucilage).

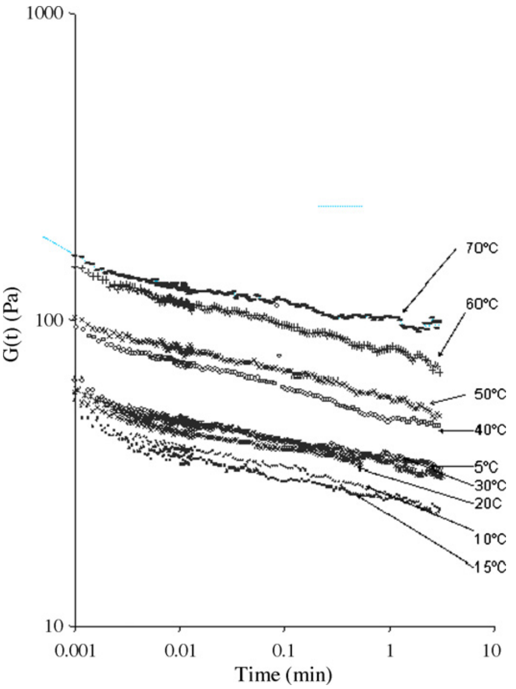

Fig. 3. Stress relaxation curves of 1% NPP solution at different temperatures (NPP: non-pectic polysaccharide from yellow mustard mucilage).

bic interactions (15–30 °C). With a larger degree of temperature increase, the relaxation curves shifted upward along the *Y* axis indicating more inter-molecular associations (30–70 °C).

Hydrophilic polymers containing a few hydrophobic units can form temporary hydrophobic association networks in aqueous media at relatively low polymer concentrations (Glass, 1989). Leibler, Rubinstein, and Colby (1991) reported that the hydrophobic groups distributed along the polymer chains might associate to minimize their exposure to water due to thermodynamic preference. When condition changes, they may form a temporary associating network which serves to reinforce the entangled network. As elucidated in our previous study, NPP molecules contain a certain amount of hydrophobic groups, mainly methyl and ethyl groups (Wu et al., 2009a, in press). This type of structure resembles some of the chemically modified cellulose derivatives such as methyl cellulose and hydroxypropyl methyl cellulose which form gels upon heating. Heat-set gels have been studied extensively although the gelation mechanism has not been completely clarified at the molecular level (Nishinari & Zhang, 2004). Jin, Zhang, Yin, and Nishinari (2006) investigated the thermal behaviour of carboxymethylated curdlan and found that G' increased at temperature from 40 to 50 °C, which is believed to relate to the swelling of the curdlan. Further increase of G' upon heating above 55 °C suggested the formation of hydrophobic interactions. X-ray diffraction studies showed that the methylcellulose gels contain crystallites of trimethyl glucose sequences (Kato, Yokoyama, & Takahashi, 1978). ^{13}C NMR studies of the high temperature gels of aqueous methylcellulose and hydroxypropyl methyl cellulose solutions revealed that the substituents on the backbone became immobile due to interchain crosslinks and phase separation (Ibbett, Philp, & Price, 1992).

Lopes da Silva, Gonçalves, and Rao (1994), after studying the influence of temperature on rheological behaviours of pectin dispersions, reported that the rheological changes of pectin dispersions due to the temperature change were the result of the effects of intermolecular forces including intermolecular hydrogen bonds, electrostatic bonds and hydrophobic interactions. Structural differences, e.g., degree of methylation, solvent properties, changes in pH, will have great influence on these intermolecular interactions. In addition, these interactions are influenced by temperature, each to a different extent and manner.

In the case of 1% NPP solution, when temperature is increased, the accelerated mobility of the molecules will increase the chance for hydrophobic groups to form crosslinks. The degree of crosssslinking might be affected by the H^+ concentration, which could produce less repulsive forces among negatively charged groups such as hydroxyl groups and carboxylate groups since NPP contains 6.9% uronic acids (Wu et al., 2009a). This could provide more chances for hydrophobic groups to bump into one another and form hydrophobic interactions. The conformation of the molecules under acidic conditions also needs to be investigated. We suspect that the acidic environment may produce a specific conformation with the hydrophobic groups more exposed which could lead to forming more hydrophobic interactions.

3.2. Relaxation behaviour in mixed NPP/GM solutions at various temperatures

The relaxation curves of 1% NPP/FG and NPP/LBG solutions at temperatures of 5, 10, 15, 30, 40, 50, 60 and 70 °C were studied. The relaxation curves at 30, 50 and 70 °C are presented in Fig. 4. Roth

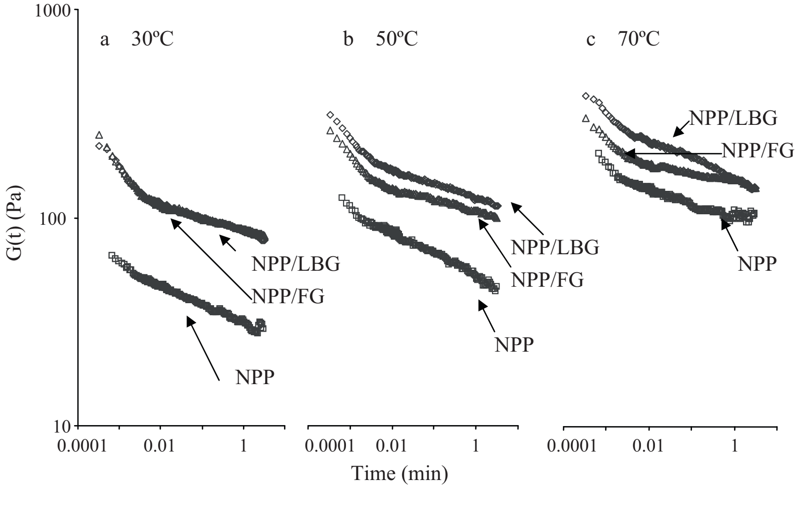

Fig. 4. Relaxation curves of mixed solutions, 1% NPP/FG and 1% NPP/LBG at various temperatures: (a) 30 °C, (b) 50 °C, and (c) 70 °C (NPP: non-pectic polysaccharide from yellow mustard mucilage; FG: fenugreek gum; LBG: locust bean gum).

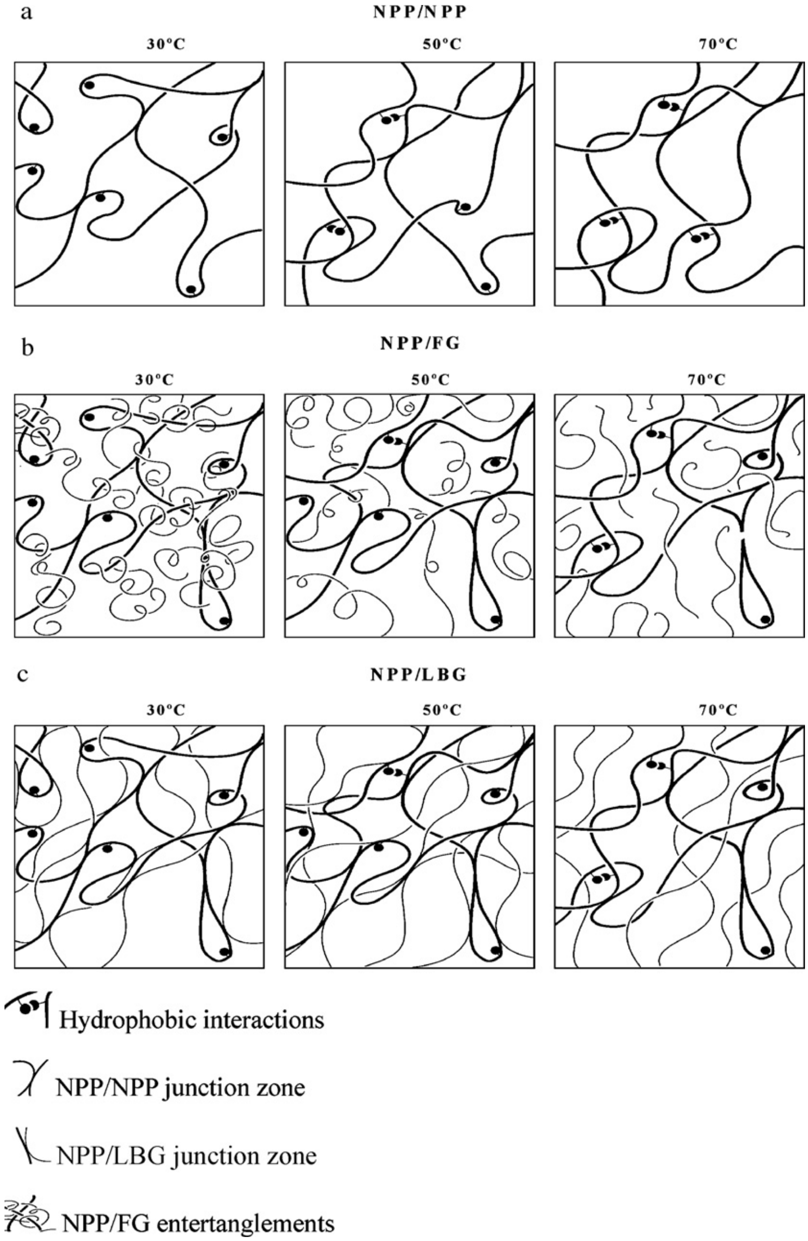

Fig. 5. Mechanism of synergistic interactions between NPP and galactomannans at different temperatures: (a) NPP/NPP; (b) NPP/FG; (c) NPP/LBG (NPP: non-pectic polysaccharide from yellow mustard mucilage; FG: fenugreek gum; LBG: locust bean gum).

et al. (2004) postulated that the relaxation time of a linear pendant chain should increase exponentially with the number of entanglements in which the chain is involved. In contrast, free linear chains trapped in a network should exhibit a power law dependence on molecular weight (de Gennes, 1979; Doi & Edwards, 1986). GMs do not form gels by themselves and therefore there should be no “structures” formed. Richardson and Ross-Murphy (1987) investigated the stress relaxation of 2% guar solutions and showed that the stress fell to baseline noise level in less than 10 s since no “structure” remained in the guar solution. However, when compared with 1% NPP solution, introducing GM into NPP solutions can make a change in the shape of stress relaxation curve as shown in Fig. 4. Molecular weight and molecular weight distribution contribute a lot to the shape and residue of the relaxation curve. A broader distribution tends to have a lower degree of relaxation (Andreassen, 1999; Ferry, 1980). In the current study, the molecular weight distributions in the mixed solutions are obviously different from that in the NPP solutions, thus the shape of the curves became different.

It can also be observed in Fig. 4 that with the increase of temperature, $G(t)$ of all solutions increased. The final values of $G(t)$ at the end of the relaxation period indicated that the mixed solutions contained more network structure than the NPP solution at the same concentration and temperature. We attribute this phenomenon to synergistic effects. At 30 °C (Fig. 4a), relaxation curves of NPP/FG and NPP/LBG solutions exhibited the same pattern. The two curves overlapped except at the first detected point ($t = 0.0001$ min), where FG showed a higher $G(t)$ value than NPP/LBG. These results are consistent with those obtained in our previous study by dynamic measurements, which showed that G' of NPP/FG was larger than that of NPP/LBG at 30 °C (Wu, Cui, Eskin, & Goff, 2009c).

In the current study, the difference in Mw between FG and LBG was relatively small (3.23×10^6 and 2.08×10^6 Da respectively) as reported in our previous paper (Wu et al., 2009c). In addition, the shapes of the relaxation curves of the two mixed solutions, NPP/FG and NPP/LBG, were almost the same at lower temperatures (5–40 °C). At increased temperatures (50–70 °C) their difference in shape can be observed, which might due to the change in cross links and entanglements related to the temperature change (Fig. 4b and c).

As elaborated in our earlier discussions (Wu et al., 2009b), synergistic interactions between NPP and GM may be due to two possible mechanisms: (i) FG forms "hyperentanglments" which penetrate within the NPP networks and thus enhance the gel strength; (ii) LBG forms junction zones with NPP through smooth regions via hydrogen bonding to improve the gelling property. At 30 °C, the molecules involved in the structure, either "hyperentanglements" or "junction zones", are relatively steady. The decrease of the $G(t)$ may be mainly due to the hydraulic pressure change as pointed out by Tang et al. (1998). This might explain the same relaxation curve of the two mixed solutions at this temperature.

At an increased temperature, 50 °C, the two mixed solutions, NPP/FG and NPP/LBG, showed different relaxation behaviours (Fig. 4b). The difference can be described by two relaxation time zones: from 0.0001 to 0.01 min, the decreasing slope of NPP/FG was steeper than that of NPP/LBG; after 0.01 min, the remaining $G(t)$ residue of NPP/LBG was consistently higher than that of NPP/FG. The difference in relaxation curves of the mixed solutions may be due to the solubility of the two GMs. FG can be readily dissolved at 50 °C while LBG can be fully dissolved at 80 °C. "Hyperentanglements" formed by FG molecules could be broken down at 50 °C, while the "junction zones" between NPP and LBG still remained at this temperature. Therefore, NPP/LBG kept more structure in the solution than NPP/FG at this temperature.

At 70 °C, more structures in the mixed solution were destroyed, except the hydrophobic interactions along the NPP backbone, which on the contrary can be enhanced by the increased temperature (Fig. 4c). Kök, Hill, and Mitchell (1999) found that LBG solutions showed remarkable increase in viscosity at temperatures above 50–80 °C and this was believed to be corresponding to solubilisation of this polymer. In NPP/FG mixtures, FG was present as free molecules interpenetrating the NPP network. In NPP/LBG solution, the hydrogen bonds were gradually broken leaving the LBG molecules free to penetrate through the NPP networks. Free linear chains trapped in a network formed by topological restrictions imposed by other chains are free to reptate (de Gennes, 1979) while the pendant chains may relax by a different mechanism (de Gennes, 1979; Doi & Edwards, 1986). In the current study, although the two types of GMs, FG and LBG, are in the category of free linear chains at this temperature, their conformation characteristics are obviously different according to our previous investigation (Wu, 2009). The conformation study by molecular modelling revealed that LBG is a much stiffer molecule than FG. Therefore, conformation might be the main cause of the different stress relaxation behaviour.

In the mixed solutions, $G(t)$ increased with the increasing temperature. However, the magnitude of increase is smaller than that of the NPP solution (Fig. 4). This difference can be explained by the concentration of NPP in mixed solutions with the NPP/GM blending ratio of 7/3, which led to a decrease of concentration of hydrophobic groups in NPP by 30%. Also, the existence of the GM chains can produce physical barriers therefore preventing NPP molecules from forming hydrophobic interactions. As indicated by Roth et al. (2004) and Ferry (1980), the stress relaxation also depends on the networks presented in the system, e.g., the concentration of cross links. Whether the existence of a different type of molecule may potentially affect the conformation of NPP thermodynamically needs further investigation.

4. Conclusion

The temperature sweep test and stress relaxation test were performed on the 1% NPP solution at various pH conditions and temperatures. The results confirmed the conclusion obtained from our earlier study that at increased temperatures, the formation of hydrophobic interactions along the NPP backbones can be enhanced. At stronger acidic conditions the gel strength also increased due to the reduced repulsive forces by introducing more H^+ ions thus providing more opportunities for the hydrophobic interactions. More investigations will be carried out to understand the impact of temperature and acid on the conformation of NPP molecules.

Stress relaxation behaviour of 1% NPP/FG and 1% NPP/LBG solutions was examined at the NPP/GM blending ratio of 7/3. The values of overall modulus ($G(t)$) in the mixed solutions were higher than those of NPP solutions due to the synergistic effects (Fig. 4). The stress relaxation curves of the NPP/FG and NPP/LBG solutions exhibited different shapes indicating different mechanisms of synergy in the two solutions. The two postulated synergistic interactions are shown schematically in Fig. 5. Fig. 5a indicates the NPP networks at different temperatures. At the low temperature (30 °C), NPP molecules formed junction zones. At a increased temperature (50 °C), the junction zones via hydrogen bonding remained largely intact while hydrophobic interactions were promoted. At a higher temperature (70 °C), the junction zones were mostly destroyed and more hydrophobic interactions were formed. Fig. 5b and c explains the difference in mechanism of NPP/FG and NPP/LBG solutions. At a lower temperature (30 °C), FG molecules formed hyperentanglements within the NPP networks (Fig. 5b); while LBG formed junction zones via H-bonding with NPP molecules (Fig. 5c). When temperature increased to a higher value (50 °C), the hyperentanglements were destroyed due to the dissolving of FG molecules at this temperature while the junction zones in NPP/LBG solution remained. At 70 °C, the junction zones between NPP and LBG molecules were destroyed and the stress modulus of NPP/FG and NPP/LBG reached the same level. The major structure remaining in both mixed solutions is from the hydrophobic interactions between NPP molecules (Fig. 5b and c). The evaluation of stress relaxation curves of the NPP/GM solutions at different temperatures provided more evidence for our previously proposed mechanism of synergistic interactions between NPP and GM.

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