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Comparative investigation of the molecular interactions in konjac gum/hydrocolloid blends: Concentration addition method (CAM) *versus* viscosity addition method (VAM)

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ABSTRACT

Investigation of molecular interactions between konjac gum and other hydrocolloids was performed by comparison of apparent measured viscosity (η_m), and those determined using the concentration addition (η_c) and viscosity addition (η_v) methods. Results could be divided into five distinct interaction levels, namely xanthan gum ($\eta_m > \eta_c > \eta_v$) > guar gum ($\eta_m \approx \eta_c > \eta_v$) > carrageenan, sodium alginate, and sodium carboxymethyl cellulose ($\eta_c > \eta_m > \eta_v$) > methylcellulose and hydroxypropyl methylcellulose ($\eta_c > \eta_m \approx \eta_v$) > gum arabic ($\eta_c > \eta_v > \eta_m$). By far the strongest, and the only true synergistic, interaction was observed with konjac/xanthan ($\eta_m > \eta_c > \eta_v$). In all other blend systems, except konjac/gum arabic, measured viscosities lay within the bounds defined by CAM and VAM, and revealed relationships for describing blend behaviour that will assist in prediction of hydrocolloid blend viscosities at finite concentrations.

1. Introduction

Konjac gum is a hydrocolloid gum derived from the tubers of *Araceae amorphophallus*, which comprises 60–70% konjac glucomannan. It is widely utilised in areas of the food industry, chemical engineering and petroleum drilling (Dhawan & Kaur, 2007). As a result of its high viscosity, konjac gum is generally added to other hydrocolloids to increase the viscosity of blended systems and improve food quality (Yaseen, Herald, Aramouni, & Alavi, 2005). However, such hydrocolloid blending can result in a wide variety of interactions, which can significantly alter (increase or decrease) specific physicochemical characteristics of the blended system compared with those of the individual colloids. Harrington and Morris (2009) found that mixing konjac gum and gelatin solutions with the same density resulted in the resolution of two liquid layers, with no synergistic interaction at all, and konjac gum and wheat starch solutions exhibited phase separation behaviour (Funami et al., 2005), however konjac gum blended with potato starch showed some synergistic interaction (Khanna & Tester, 2006). Thus, investigation of the interactions between mixtures of konjac gum and other hydrocolloids is of significant interest and value with respect to food colloid complex formulation development, food system thickening and stabilisation, and other areas such as fluid transportation. Likewise, the ability to accurately predict the viscosities of such blends would be highly advantageous.

Apparent viscosity and gelation strength are routinely utilised to assess synergistic interactions in mixed hydrocolloid systems (Morris, 1984, 1995). Generally, if one composite shows higher viscosity or gelation strength than both its constituent hydrocolloids at a particular concentration, it could be considered that they synergistically interact with each other. However, this is an over simplification, since viscosity and concentration are not linearly related, but by a power law. It is therefore often inappropriate for use of a concentration addition method, and therefore use of a viscosity addition method is required (Morris, 1984, 1995). Differences between the concentration addition and viscosity addition methods, and the associated mathematical relationships/equations, are detailed and discussed subsequently (Section 3.1).

A neutral polysaccharide (guar gum), four ionic polysaccharides (xanthan, carrageenan, sodium alginate, and sodium carboxymethyl cellulose), two weakly hydrophobic polysaccharides (hydroxypropyl methylcellulose, and methylcellulose) and an emulsifying polysaccharide with hydrophobic groups (gum arabic) were blended with konjac gum, respectively. By determination of the apparent viscosity of the component and blend hydrocolloid

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systems, systematic assessment of the interaction between konjac gum and these eight other hydrocolloids was performed, and these results were compared with calculated values from the concentration addition method (CAM) and the viscosity addition method (VAM).

2. Materials and methods

2.1. Materials

The hydrocolloids detailed previously were sourced commercially: konjac gum (Hubei Shiyan Huaxianzi Konjac Production Co. Ltd, China); xanthan, guar gum, and carrageenan (Heinan Shijimei Food Additives Co. Ltd, China); sodium alginate, and gum arabic (Sinopharm Chemical Reagent Co. Ltd, China); sodium carboxymethyl cellulose (degree of substitution, DS = 0.8, Tianjin Kermel Chemical Reagent Co. Ltd, China); hydroxypropyl methylcellulose ($DS_{methoxy} = 0.73$, $DS_{hydroxypropyl} = 0.46$) and methylcellulose (DS = 1.8, Shandong Heda Co. Ltd, China).

Sodium alginate, gum arabic, and sodium carboxymethyl cellulose were chemical grade, whilst all others were food grade. All other reagents/chemicals detailed in subsequent sections were of suitable laboratory grade.

2.2. Hydrocolloid solution preparation

Individual hydrocolloids were dispersed in distilled water (0.2, 0.4, 0.6, 0.8, and 1%, w/w, 100 mL), mechanically stirred (2 h at 200 rpm); left to stand for 1 h, and the resulting apparent solution viscosity measured at 25 °C, as detailed below. The carrageenan solution required heating (80 °C) to achieve solubilisation (Nussinovitch, Corradini, Normand, & Peleg, 2000), and the hydroxypropyl methylcellulose and methylcellulose were added to hot water (80 °C) to avoid aggregation (Li, Xu, & Xie, 2006). Konjac gum was separately mixed with the eight other hydrocolloids as detailed below, with blend ratios set at approximately 1:4, 2:3, 3:2, and 4:1, and the total hydrocolloids concentration fixed (at 1%, w/w, 100 mL). Xanthan, guar gum, sodium alginate, sodium carboxymethyl cellulose, and gum arabic could be mixed with konjac gum in dry powder form and prepared as detailed above, whereas carrageenan required separate dissolution in distilled water with subsequent heating at 80 °C (Nussinovitch et al., 2000), and then addition of konjac gum powder. Likewise, hydroxypropyl methylcellulose and methylcellulose were respectively dispersed in hot water (80 °C) with stirring and then mixed with konjac gum powder (Li et al., 2006).

2.3. Viscosity measurements

Viscosity measurements were performed using a NDJ-8S rotation viscometer (Shanghai Tianping Equipment and Instrument Co. Ltd, China). All viscosities were measured at 25 ± 0.5 °C in triplicate using a No. 4 rotator at 12 rpm, except for individual gum arabic solutions, which were measured using a No. 1 rotator at 60 rpm (due to their low viscosity).

3. Results and discussion

3.1. Mathematical relationships between viscosity and concentration

At a fixed temperature the relationship between the viscosity (η) and concentration (C) for a hydrocolloid solution can be described by the power law Eq. (1), where α is the viscosity coefficient, and β is the viscosity index (Morris, 1984, 1995).

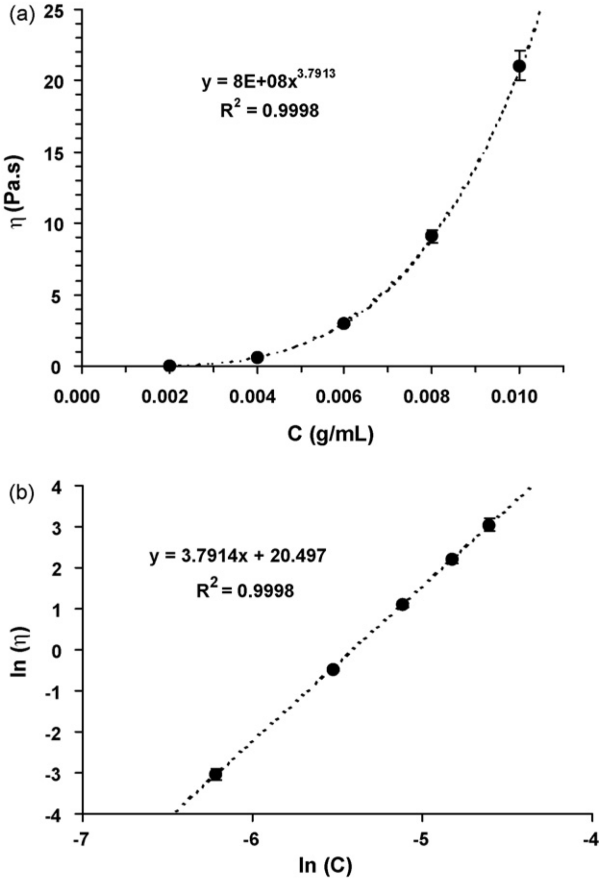

Fig. 1. (a) Power law, and (b) linear relationships between viscosity (η) and concentration (C), for konjac solutions of increasing concentration (0.2–1.0%, w/w).

$$\eta = \alpha C^{\beta} \quad (1)$$

In the ‘concentration addition method’ (CAM), the viscosity of a composite solution (η_c) composed of two hydrocolloids can be calculated using Eq. (2), where x is the fraction of hydrocolloid 1 in the composite and α_1 and β_1, and α_2 and β_2 are the viscosity coefficients and viscosity indexes for hydrocolloid 1 and hydrocolloid 2, respectively.

$$\eta_C = x\alpha_1 C_1^{\beta_1} + (1-x)\alpha_2 C_2^{\beta_2} \quad (2)$$

In the ‘viscosity addition method’ (VAM), the viscosity of a composite solution (η_v) composed of two hydrocolloids can be calculated using Eq. (3), where x is the fraction of hydrocolloid 1 in the composite and α_1 and β_1, and α_2 and β_2 are the viscosity coefficients and viscosity indexes for hydrocolloid 1 and hydrocolloid 2, respectively.

$$\eta_v = \alpha_1 (xC_1)^{\beta_1} + \alpha_2 [(1-x)C_2]^{\beta_2} \quad (3)$$

3.2. Viscosity coefficients and viscosity indexes for individual hydrocolloids

The determined average apparent viscosities (η) for the individual hydrocolloid solutions (analysed in triplicate) were plotted as a function of concentration (C) over the investigated range of 0.2–1.0% (w/w), and a power law ‘trendline’ applied to the data points (using Microsoft Excel). An example curve for konjac gum is presented (Fig. 1(a)), and the trendline equation provides values for the viscosity coefficient (α) and viscosity index (β). All replicate viscosity measurements demonstrated % variation

(100 × [standard deviation/arithmetic mean]) values of <6%. The relationship between apparent viscosity and concentration can also be presented linearly, by plotting $\ln(\eta)$ *versus* $\ln(C)$, giving a straight line of gradient β, and y-intercept $\ln(\alpha)$ (Fig. 1(b)). The determined values for α and β are obviously dependent upon the viscosity and concentration units utilised, however, in the case of such determinations viscosity is generally quoted in Pa s and concentration in g/mL.

All of the data obtained for the hydrocolloids investigated (except gum arabic) correlated reasonably well with power law Eq. [1] (correlation coefficients, $R = 0.993–1.000$) over the investigated concentration range of 0.2–1.0% (w/w). The obtained apparent viscosity values for gum arabic were too low (all <5 mPa s) over the investigated concentration range to obtain correlatable results. The resultant viscosity coefficient (α), viscosity index (β), and regression analysis correlation coefficient (R) values for the individual hydrocolloid solutions (with the exception of gum arabic), determined as detailed above, are presented (Table 1). Some slight differences between the results from power law (e.g. Fig. 1(a)) and linear (e.g. Fig. 1(b)) plots were observed.

3.3. *Measured and calculated viscosity values for hydrocolloid blends*

The viscosity curves as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL at 25 ± 0.5 °C) for konjac/other hydrocolloid blends are presented in Figs. 2–4. 'Measured blend' (--▲--), refers to the averaged results from triplicate measurements performed on the konjac/other hydrocolloid blends. 'VAM (measured)' (--■--), refers to the viscosity determined by simple addition of the relative individual component viscosities for the pure hydrocolloids, e.g. in the case of konjac/guar 3:2 blend, this is the viscosity of the konjac 0.006 g/mL solution added to the viscosity of the guar 0.004 g/mL solution (total hydrocolloid concentration of 0.010 g/mL). 'VAM (calculated)' (--○--), refers to the viscosity values calculated using the viscosity addition method (Eq. [3]) and the viscosity coefficient (α) and viscosity index (β) values determined previously for the individual hydrocolloids from power law trendlines (Table 1). 'CAM (measured)' (—■—), refers to the viscosity determined by simple addition of the relative percentages of the individual component viscosities for the pure hydrocolloids, e.g. in the case of konjac/guar 3:2 blend, this is 60% of the viscosity of the konjac 0.010 g/mL solution added to 40% of the viscosity of the guar 0.010 g/mL solution. 'CAM (calculated)' (—○—), refers to the viscosity values calculated using the concentration addition method (Eq. [2]) and the viscosity coefficient (α) and viscosity index (β) values determined previously for the individual hydrocolloids from power law trendlines (Table 1). In all cases, CAM (measured) ≈ CAM (calculated), and VAM (measured) ≈ VAM (calculated).

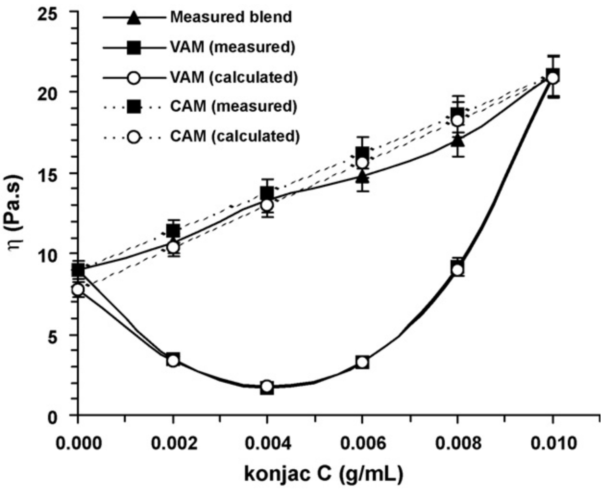

Fig. 2. Konjac/neutral polysaccharide (guar gum) blend viscosity as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL), as determined by measured and calculated viscosity values.

A synergistic effect is observed if the 'measured blend' viscosity (η_m) is greater than both the CAM (η_c) and VAM (η_v) measured and calculated viscosity values ($\eta_m > \eta_c > \eta_v$). Conversely, if $\eta_m < \eta_v < \eta_c$ then blending has resulted in a weakening effect. In the absence of any intermolecular interaction/binding, $\eta_m \approx \eta_v$. Therefore if $\eta_c > \eta_m > \eta_v$ then there is some evidence of intermolecular interaction/binding.

3.3.1. *Viscosity of konjac/neutral polysaccharide blends*

The viscosity curves as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL at 25 ± 0.5 °C) for konjac/guar gum blends is presented in Fig. 2. The 'measured blend' viscosities for the konjac/guar blends were very similar to the CAM (measured and calculated) viscosity values (i.e. $\eta_m \approx \eta_c > \eta_v$), therefore there was no synergistic interaction with respect to the CAM results. However, with respect to the VAM results, a konjac/guar 2:3 blend ratio gave the maximum viscosity increase (>7.5 *x*), with a blend series average increase of >4 *x*.

3.3.2. *Viscosity of konjac/ionic polysaccharide blends*

The viscosity curves as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL at 25 ± 0.5 °C) for konjac/xanthan, konjac/carrageenan, konjac/sodium alginate, and konjac/sodium carboxymethyl cellulose blends are presented in Fig. 3(a)–(d), respectively.

Konjac/xanthan blends clearly demonstrated a significant synergistic interaction ($\eta_m > \eta_c > \eta_v$). With respect to the CAM results,

Table 1
Hydrocolloid solution viscosity parameters determined using power law (e.g. Fig. 1a), and linear (e.g. Fig. 1b) relationships between viscosity (η) and concentration (C).

Hydrocolloid	Power law graph[a]		Ln graph[b]		
	α	β	α	β	R
Konjac gum	8.00×10^8	3.7913	7.98×10^8	3.7914	0.9999
Xanthan gum	2.20×10^5	2.1743	2.20×10^5	2.1748	0.9941
Guar gum	4.00×10^8	3.8545	4.32×10^8	3.8552	0.9991
Carrageenan	9.69×10^2	1.9917	9.70×10^2	1.9921	0.9969
Sodium alginate	1.21×10^3	1.7103	1.21×10^3	1.7103	0.9965
Sodium carboxymethyl cellulose	4.82×10^2	1.5775	4.80×10^2	1.5770	0.9938
Hydroxypropyl methylcellulose	3.80×10^5	2.9207	3.89×10^5	2.9257	0.9926
Methylcellulose	2.86×10^5	3.0814	2.89×10^5	3.0834	0.9939

α – viscosity coefficient; β – viscosity index; R – regression analysis correlation coefficient.

[a] Calculated from power law trendline (as in Fig. 1a).

[b] Calculated from ln graph linear trendline (as in Fig. 1b).

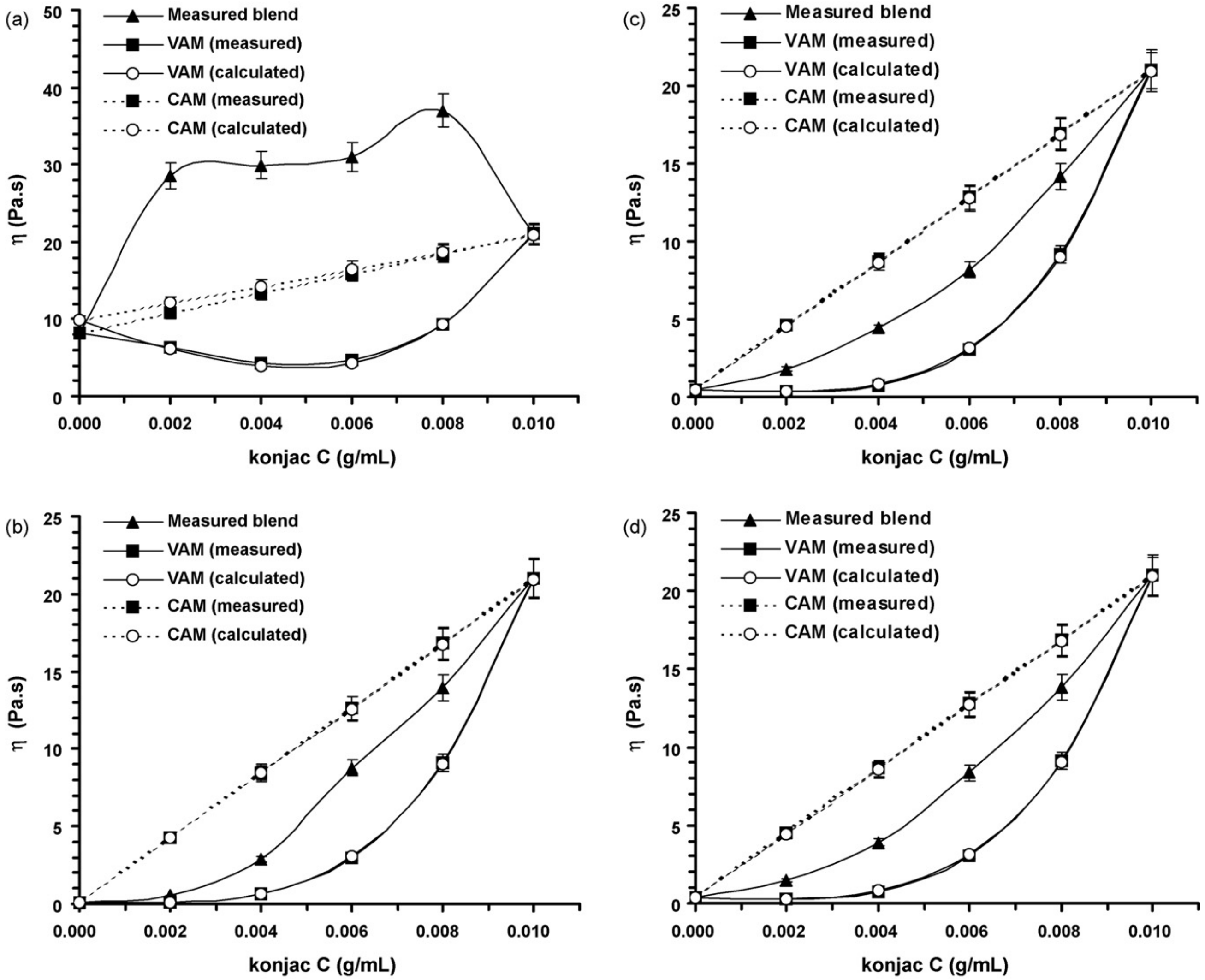

Fig. 3. Konjac/ionic polysaccharide blend viscosity as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL), as determined by measured and calculated viscosity values: (a) xanthan, (b) carrageenan, (c) sodium alginate, and (d) sodium carboxymethyl cellulose.

a konjac/xanthan 1:4 blend ratio gave the maximum viscosity increase (>2.5 *x*), with a blend series average increase of >2 *x*. With respect to the VAM results, a konjac/xanthan 2:3 blend ratio gave the maximum viscosity increase (>7 *x*), with a blend series average increase of >5.5 *x*. Such strong synergistic effects indicate that some other specific interactions, besides Van der Waals forces, must be occurring between the two different hydrocolloid polymer macromolecules, however the mechanisms involved in such interactions are not clear.

The best known, and most controversial, examples of synergism involve interaction of galactomannans, konjac glucomannans and related plant polysaccharides with xanthan or with algal polysaccharides in the agar/carrageenan series (Dea & Morrison, 1975; Morris, 1995). Two common features of these systems are that the plant polysaccharides all share the same 1,4-diequatorial linkage geometry (so that their ordered structures in the condensed phase are flat, extended ribbons), and that the other components all undergo thermoreversible disorder–order transitions under hydrated conditions (Morris, 1995).

Konjac glucomannan can undergo self-association in aqueous solution, and mixing with xanthan at low temperature may promote self-association within konjac glucomannan molecules and intermolecular interactions with xanthan molecules. It has been suggested that when xanthan chains are in the disordered state, intermolecular association involves attachment of plant polysaccharide to the backbone of the xanthan molecule forming a novel 6-fold helix under favourable conditions (Brownsey, Cairns, Miles, & Morris, 1988; Cairns, Miles, & Morris, 1986; Cairns, Miles, Morris, & Brownsey, 1987; Chandrasekaran, Janaswamy, & Morris, 2003; Morris, 1992). In the case of these investigations, the mixing is carried out with xanthan in the partially ordered form, whereas if the mixing were carried out above the helix–coil transition temperature of xanthan then the effect would probably be much larger. Other studies have shown that the xanthan side chains and ordered portions of the macromolecular complex in solution are important, and that a konjac/xanthan ratio of 45:55 is the preferred stoichiometry for maximum viscosity (Paradossi, Chiessi, Barbiroli, & Fessas, 2002).

Konjac glucomannan and xanthan gum mixed solutions have been used to develop delivery systems capable of maintaining physical integrity and drug release control (Alvarez-Manceñido, Landin, Lacik, & Martínez-Pacheco, 2008).

Konjac/carrageenan, konjac/sodium alginate, and konjac/sodium carboxymethyl cellulose blends demonstrated similar viscosity profiles (with $\eta_c > \eta_m > \eta_v$, Fig. 3(b)–(d), respectively). With respect to the VAM results, konjac/carrageenan, konjac/sodium alginate, and konjac/sodium carboxymethyl cellulose blends demonstrated maximum viscosity increases at blend ratios

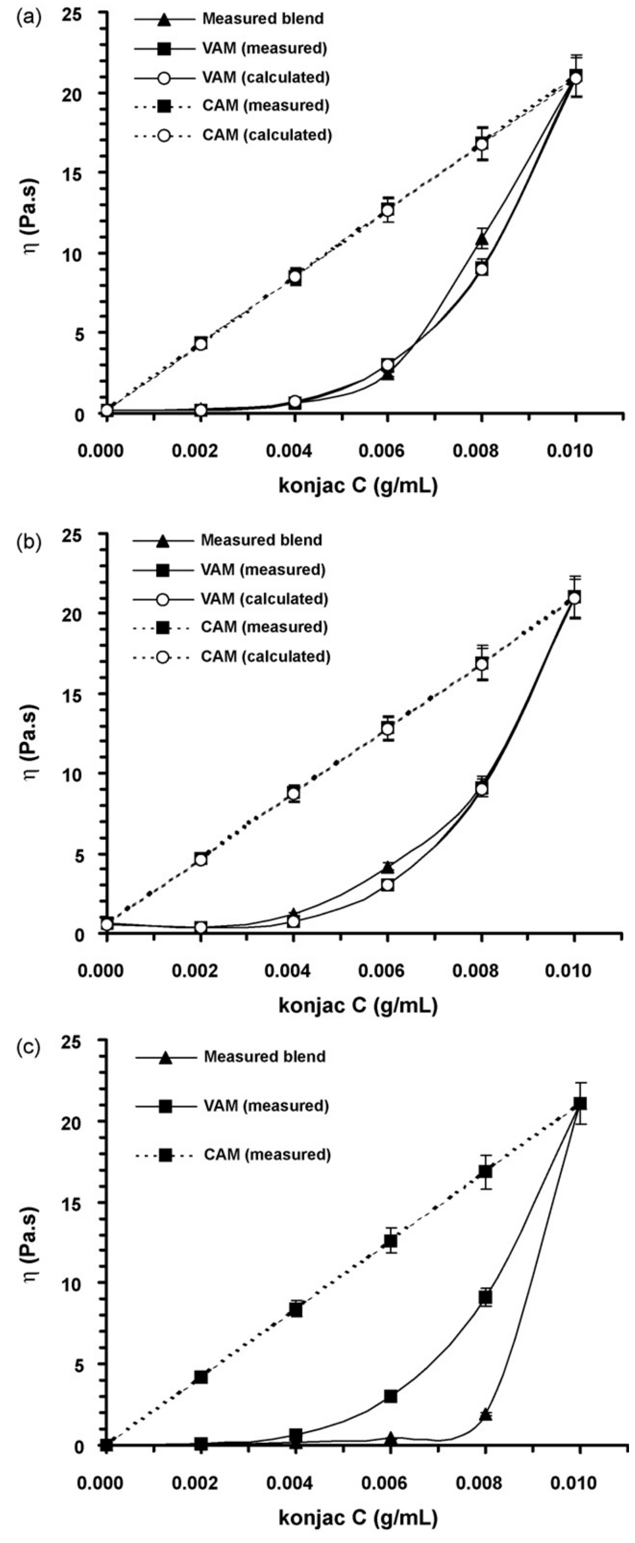

Fig. 4. Konjac/hydrophobic polysaccharide blend viscosity as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL), as determined by measured and calculated viscosity values: (a) methyl cellulose; (b) hydroxypropyl methylcellulose, and (c) gum arabic.

of 1:4 (>4.5 x), 2:3 (>5.5 x), and 1:4 (>5 x), respectively, with blend series average increases of >3 x, >3.5 x, and >3.5 x, respectively.

Konjac/carrageenan gum mixing system is widely applied in the food industry, for example as jelly and ice cream stabilisers, however their intermolecular interaction mechanisms have not been fully elucidated. Studies on the association between konjac glucomannan and κ-carrageenan have shown that interaction occurs only when the carrageenan is in its ordered helical form, and it has been proposed that the molecules associate through hydrogen bonding once the ordered structure has formed (Williams, 2009).

Previous studies have shown that synergistic viscosity increases in konjac glucomannan/carboxymethyl cellulose solutions (Cheng, Abd Karim, Norziah, & Seow, 2002) and blend films (Yang & Zhu, 2007) can be attributed to increased levels of hydrogen bonding.

3.3.3. *Viscosity of konjac/hydrophobic polysaccharide blends*

The viscosity curves as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL at 25 ± 0.5 °C) for konjac/methyl cellulose, konjac/hydroxypropyl methylcellulose, and konjac/gum arabic blends are presented in Fig. 4(a)–(c), respectively.

The ‘measured blend’ viscosities for the konjac/methyl cellulose and konjac/hydroxypropyl methylcellulose blends were very similar to the VAM (measured and calculated) viscosity values (i.e. $\eta_c > \eta_m \approx \eta_v$), therefore there were only very small (if any) interactions with respect to the VAM results (Fig. 3(a) and (b), respectively). With respect to the VAM results, konjac/methyl cellulose and konjac/hydroxypropyl methylcellulose blends demonstrated maximum viscosity increases at blend ratios of 1:4 (>1.75 x), and 2:3 (>1.5 x), respectively, with blend series average increases of $\sim$ 1.3 x, and $\sim$ 1.2 x, respectively. Similar results were obtained with konjac glucomannan–methyl cellulose hydrocolloid films (Li et al., 2006). Dynamic viscoelastic and fluorescence probes have been used to show that the hydrophobic interactions in konjac gum are similar to those in methyl cellulose and hydroxypropyl cellulose (Chen, 2009; Zhu, 2008). Yin, Zhang, Huang, and Nishinari (2008) showed that the konjac glucomannan backbone had weak hydrophobic properties.

The viscosity curves as a function of relative component concentration (total hydrocolloid concentration maintained at 0.01 g/mL at 25 ± 0.5 °C) for konjac/gum arabic blends are presented in Fig. 4(c). The ‘measured blend’ viscosities for the konjac/gum arabic blends were lower than both the CAM and VAM (measured) viscosity values (i.e. $\eta_c > \eta_v > \eta_m$), therefore gum arabic had a negative (weakening) effect on viscosity. CAM and VAM (calculated) could not be determined since, as stated previously, the viscosities of pure gum arabic solutions were too low over the tested concentration range (0.002–0.010 g/mL) to facilitate determination of the viscosity coefficient (α) and viscosity index (β) values. With respect to the VAM (measured) results, konjac/gum arabic blends demonstrated average viscosity increases of $\sim$0.5 x, and $\sim$0.05 x with respect to CAM results.

Gum arabic is highly water-soluble (up to 50%, w/w), has extremely low viscosity in comparison to linear polysaccharides, due to its dense branched structure, and has both hydrophilic and hydrophobic groups, giving it lipophilic emulsion characteristics (Giannouli, Richardson, & Morris, 2004). Ahmed, Ramaswamy, and Ngadi (2005) reported that gum arabic can significantly reduce the rheological properties of both guar and xanthan gum. The specific mechanisms of gum arabic/konjac glucomannan interaction require further investigation.

4. Conclusions

The results of the assessment of the molecular interactions between konjac gum and the other eight hydrocolloids investigated can be divided into five distinct levels of interaction, namely xanthan gum > neutral non-ionic polysaccharide (guar gum) > ionic polysaccharides (carrageenan, sodium alginate, and sodium carboxymethyl cellulose) > hydrophobic polysaccharides (methylcellulose and hydroxypropyl methylcellulose) > emulsifying hydrophobic polysaccharide (gum arabic).

By far the strongest interaction, and the only true synergistic interaction, was observed with konjac/xanthan blends ($\eta_m > \eta_c > \eta_v$). This was followed by konjac/guar gum blends, which demonstrated viscosity curves very similar to the concentration addition method (CAM) results ($\eta_m \approx \eta_c > \eta_v$). The increased viscosities of both of these blend systems could be attributed to enhanced hydrogen bonding. Konjac/carrageenan, sodium alginate, and sodium carboxymethyl cellulose blends all had similar viscosity curves ($\eta_c > \eta_m > \eta_v$), whilst konjac/methyl cellulose and hydroxypropyl methylcellulose blends demonstrated viscosity curves very similar to the viscosity addition method (VAM) results ($\eta_c > \eta_m \approx \eta_v$). The former group therefore experienced some enhanced interaction, whilst the latter group only had similar relatively weak intermolecular forces (such as Van der Waals forces). Finally, konjac/gum arabic blends demonstrated greatly reduced viscosities ($\eta_c > \eta_v > \eta_m$), possibly due to gum arabic disrupting konjac hydrogen bonding/hydrophobic interactions.

In summary, in all investigated blend systems, except konjac/xanthan and konjac/gum arabic, the measured viscosities lay within the bounds defined by the CAM and VAM methods. With the exception of gum arabic, the investigations revealed interesting blending laws for describing the behaviour of the mixtures where the interactions are indirect. In the absence of intermolecular binding, interactions presumably reflect effects such as competition for solvent and differential swelling, which alter the effective concentration, or concentration dependence of the components in the mixtures.

Adopting the viscosity addition method (VAM) alone identifies more interactions as being potentially synergistic, compared with using the concentration addition method (CAM), however there is considerable speculation as to whether or not all such identified interactions are indeed synergistic. Both methods are of value and can easily be utilised in conjunction. The detailed evaluation of interactions between konjac gum and other complex polysaccharides is required in order to shed further light on the molecular interaction mechanisms involved, and will assist in the ability to predict such system blend viscosities at finite concentrations.

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