



# Comparative assessment of dynamic oscillatory measurements on network development and mechanical spectra of gelatine or gellan in maize starch–egg white composite gels



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## ABSTRACT

Single component gels (SCG) were formed from gelatine (Glt), gellan (Gll), maize starch (MS) or egg white (EW), while binary component gels (BCG) and tertiary component gels (TCG) were formed by mixing Glt or Gll with MS or/and EW. All gels were evaluated by dynamic oscillatory test. Each type of SCG exhibited distinct network formation profiles. The effects of Glt or Gll proportions on the network formation profiles of BCG and TCG were investigated using mixture design experiments. Glt and Gll yielded composite gels that were remarkably different in terms of network formation profiles. In all [MS:Glt/Gll]-BCG and TCG systems, the network structures were developed during cooling suggesting that Glt and Gll being the main components contributing to network formation. However, for [EW:Glt/Gll]-BCG, component with higher ratios governed the gelling mechanism, while the component with lower proportions acted as an inactive filler within the network.

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## 1. Introduction

Gellan (Gll) has been proposed as one of the gelatine (Glt) alternatives in food applications (Morrison, Sworn, Clark, Chen, & Talashek, 1999). It was hypothesised that Gll would reveal the same gelling effects as Glt in composite gel systems, as Glt and Gll are both helix-forming biopolymers that form gels on cooling. However, Glt gels are soft, elastic, translucent and thermoreversible (Glücksman, 1969; Ledward, 2000), whilst low-acyl Gll gels are clear, firm to touch, brittle and usually non-thermoreversible (Huang, Kennedy, Li, Xu, & Xie, 2007; Williams & Philips, 2003). The strength and texture of Gll gels are dependent on ionic strength, while those of Glt gels depend more on the concentration of Glt (Lee et al., 2003; Morris, Nishinari, & Rinaudo, 2012; Panouillé & Larreta-Garde, 2009; Pérez-Campos, Chavarría-Hernández, Tecante, Ramírez-Gilly, & Rodríguez-Hernández, 2012). A recent study by Foo, Liong, and Easa (2013) reported that Glt gel is several times harder than Gll gel, but its breakdown was the fastest due to its susceptibility to melt at 37 °C.

In the food industry, a convenient approach to produce novel food products with the desired functional properties is by manipulating the combinations of different biopolymers (e.g., starch and protein). A broad range of characteristics would be expected owing to the diversity and complexity of food ingredients available. Therefore, it is critical to understand the interactions of various components within the system in order to achieve the targeted properties (Elgadir et al., 2012). To our knowledge, very few studies have been performed on comparative assessment of composite gels prepared from Glt and Gll. In our earlier study, composite gels prepared from Glt and Gll with maize starch (MS) and/or egg white (EW) exerted different effects on textural, rheological and microstructure properties in both binary and ternary composite gel systems (Tan, Foo, Liong, & Easa, 2014a, 2014b). It was hypothesised that these differences could be due to different network formation profiles during gel formation.

Dynamic rheological tests have been used to provide valuable information on molecular interactions during gel formation and development during ageing. The response of a material to sinusoidally varying of stress or strain during oscillation is recorded as dynamic moduli (Ikeda & Foegeding, 2003). The two moduli are defined as: 1) in-phase stress/strain or storage modulus ( $G'$ ); and 2) out-of-phase stress/strain or loss modulus ( $G''$ ).  $G'$  is a measure of

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the energy stored in the material and subsequently recovered per cycle of oscillation. It is taken as an indication of solid or elastic characteristic of a material and the magnitude of  $G'$  is dependent upon the molecular rearrangements occurred in the sample. On the other hand,  $G''$  is a measure of the energy dissipated as heat per cycle of oscillation and is taken as an indication of liquid or viscous behaviour. Another parameter that is commonly used to indicate the physical behaviour of a sample is loss tangent ( $\tan \delta$ ). It is a ratio of energy lost to energy stored for each sinusoidal deformation cycle, i.e.,  $\tan \delta = G''/G'$ . It can also be used as an indication of the relative contributions of viscous and elastic components in a viscoelastic material (Burey, Bhandari, Rutgers, Halley, & Torley, 2009; Ikeda & Foegeding, 2003).

The objective of this study was to comparatively study the influence of ingredient composition on the network formation profiles of Glt and GII in composite gel systems containing MS or/and EW. This study investigates the network formation profiles of composite gel systems by following the gel formation profiles in order to elucidate the interactions between each component. To further understand the mechanical properties of the gels studied, it is interesting to reveal the real time network formation of the gels using a small deformation oscillation test. The time–temperature profile used to monitor the network development was in accordance with the gel preparation procedures with the adjustment of setting in time.

## 2. Materials and methods

### 2.1. Materials

Gelatine (source: bovine, bloom strength 160, pH 5.3) was obtained from Halagel Sdn. Bhd., Kedah, Malaysia. Low-acyl gellan was supplied by Fluka Chemical Corp., Ronkonkoma, USA. Maize starch was purchased from Roquette Freres S.A., Lestrem, France. Egg albumen powder (instant high gel, EAP-HG) was purchased

from Belovo S.A., Bastogne, Belgium. This high-gelling egg albumen powder contains protein and moisture contents of 82.5% and 5.9%, respectively.

### 2.2. Biopolymer stock preparation

The four selected biopolymers employed in this section were of common ingredients used for a gelling purpose in food products, namely MS, EW, Glt and GII. Single component gels (SCG) were formed from Glt, GII, MS or EW, while binary component gels (BCG) and tertiary component gels (TCG) were formed by mixing Glt or GII with MS or/and EW powders (Table 1). SCG with specific targeted modulus values ( $24,000 \text{ N m}^{-2}$ ) were used as a benchmark against composite gels to yield free-standing gels (Foo et al., 2013). During preliminary work, MS and EW required 13% (w/w) and 9% (w/w) of biopolymers, respectively to yield SCG with modulus values of in close proximity to  $24,000 \text{ N m}^{-2}$ . However, the modulus values for Glt and GII deviated from the targeted modulus as the concentrations of biopolymers were adjusted in order to prepare gels that suited the whole experimental conditions, where free-standing gels were necessary for the analyses. For Glt-SCG, the amount of biopolymer selected was 11% (w/w) although the modulus obtained ( $\sim 19,000 \text{ N m}^{-2}$ ) was lower than the targeted value. However, it was not suitable to raise the amount of Glt for composite gel formulations due to the effect of limited water for a complete biopolymer hydration. As for GII-SCG, the selected amount of biopolymer (2.5%, w/w), yielded SCG with modulus ( $\sim 35,000 \text{ N m}^{-2}$ ) that exceeded the target values. However, it was not suitable to prepare GII-SCG with the targeted modulus value ( $24,000 \text{ N m}^{-2}$ ) since below 2.5% (w/w) of GII concentration no free-standing composite gels (GII-BCG and GII-TCG) could be formed. Thus, the concentrations of MS, EW, Glt and GII were 13, 9, 11 and 2.5% (w/w), respectively.

The stock biopolymer (Glt, GII, MS and EW) dispersions were prepared separately according to the method described by Tan et al. (2014a). The individual biopolymers were dispersed in distilled water and were then left overnight at  $15^\circ\text{C}$  (Sanyo Electric, MIR-254, Moriguchi, Japan) to ensure complete swelling. Glt and GII dispersions were dissolved by heating the dispersions at  $80^\circ\text{C}$  in a water bath (Memmert, WB22, Schwabach, Germany) and stirred constantly until clear solutions were formed. MS slurry and EW solution were vacuum-degassed using a diaphragm vacuum pump (Vacuumbrand, ME 2C, Wertheim, Germany) for 20 min under continuous stirring on a magnetic stirrer (Heidolph Instruments, MR Hei-Tec, Schwabach, Germany) to avoid bubble formation. All the biopolymer stock solutions were kept at  $50^\circ\text{C}$  in a water bath prior to blend preparation as depicted in Table 1.

### 2.3. Dynamic oscillatory test

The gelation profiles of the designated blends were investigated by dynamic oscillatory measurements (Rocha, Teixeira, Hilliou, Sampaio, & Gonçalves, 2009). The tests were performed on a controlled-stress rheometer (TA Instruments, AR 1000-N, Leatherhead, UK) using a 40 mm steel parallel plate with solvent trap geometry. The designated blend was transferred onto the Peltier plate, which was preheated at  $50^\circ\text{C}$ . The measuring gap between the plates was 1 mm and the exposed edge was coated with a thin layer of silicon oil to prevent moisture loss. In addition, a solvent trap cover was also used so that the atmosphere under the cover was saturated with water. The sample was allowed to relax and equilibrate at the initial temperature ( $50^\circ\text{C}$ ) for 5 min prior to assess of its rheological properties.

The evolution of dynamic moduli was used to determine the gelation profile for each blend. Thus, the temperature ramp was set

**Table 1**  
Formulations of designated blends for SCG, BCG and TCG.

Samples	Component proportions <sup>a</sup>			
	A: MS	B: EW	C: Glt	D: GII
SCG				
MS	1.00	–	–	–
EW	–	1.00	–	–
Glt	–	–	1.00	–
GII	–	–	–	1.00
BCG				
[67MS:33EW]	0.67	0.33	–	–
[50MS:50EW]	0.50	0.50	–	–
[33MS:67EW]	0.33	0.67	–	–
[67MS:33Glt]	0.67	–	0.33	–
[50MS:50Glt]	0.50	–	0.50	–
[33MS:67Glt]	0.33	–	0.67	–
[67MS:33GII]	0.67	–	–	0.33
[50MS:50GII]	0.50	–	–	0.50
[33MS:67GII]	0.33	–	–	0.67
[67EW:33Glt]	–	0.67	0.33	–
[50EW:50Glt]	–	0.50	0.50	–
[33EW:67Glt]	–	0.33	0.67	–
[67EW:33GII]	–	0.67	–	0.33
[50EW:50GII]	–	0.50	–	0.50
[33EW:67GII]	–	0.33	–	0.67
TCG				
[MS:EW:Glt] <sup>b</sup>	0.33	0.33	0.33	–
[MS:EW:GII] <sup>b</sup>	0.33	0.33	–	0.33

Notes: SCG = single component gel, BCG = binary composite gel, TCG = ternary composite gel, Glt = gelatine, GII = gellan, MS = maize starch, EW = egg white.

<sup>a</sup> A + B + C + D = 1 or 100%.

<sup>b</sup> Composite gels with equal proportions of each component.

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