RHEOLASER MASTER

Optimal gelling agent concentration

Accurate and efficient gelation studies

INTRODUCTION

In many food systems gels can be found, either for their unique properties to stabilize or for their textures. Especially, polymers and biopolymers are used for gel formation. Typical questions for formulators facing a new unknown gelling agent are:

A) What is the minimum concentration of gelling agent needed to form a gel?
B) What is the gel point for a given concentration?
C) What is the gel strength of each gel?
D) What time is needed to match a reference elasticity?

To answer these questions, a formulator must study many samples, which is time-consuming. Thanks to Rheolaser, 6 samples can be studied simultaneously, reducing significantly the time to characterize an unknown sample.

Gelling agent **Concentration** **Gel point**

HOW IT WORKS

Rheolaser Master is based on Diffusing Wave Spectroscopy (DWS), a multiple light scattering technique. Light is backscattered by scatterers in the sample. The microstructure motion inside the sample (droplets, crystallites, etc.), creates an interference pattern (Speckle Image). Variation of this image in time is directly related to the mobility of the scatterers. (Figure 1a). The faster the Speckle Image changes in time, the higher the mobility of the microstructure.

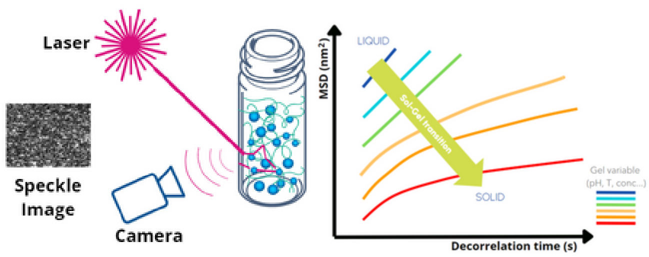

Figure 1. Schematical representation of the measurement set-up.

By mathematical treatment, Mean Square Displacement (MSD) curves are obtained (Figure 1b), which contain the viscoelastic information. Straight lines (blue) indicate a purely viscous behavior of the sample. The viscosity can be calculated as the Macroscopic Viscosity Index related to the slope in a double-linear scale. Curves with a plateau (red) indicate gel-like or solid-like behavior. The elasticity index is calculated at the elastic plateau. The lower the plateau, the higher the elasticity.

SAMPLES

Milk samples were acidified with different concentrations of glucono-δ-lactone (GDL). This cyclic lactone is hydrolyzed in contact with water and lowers the pH of the sample. Milk will coagulate when reaches a pH lower than pH 5. The acidification kinetic is highly dependent on the GDL concentration. Samples were measured at 42°C

RESULTS

A) What is the minimum gelling agent concentration?

A minimum of 12 samples is necessaire for this experiment. Thanks to the 6 measurement position, this can be done in 2 runs, with a minimum operator time. After the measurement, MSD curves were chosen (e.g. at the same time or the end of the experiment) to give Figure 2-I.

The MSD curves in the top left are those with low concentrations of gelling agent. They are linear, which indicates liquid behavior. With increasing concentration, gels are formed, leading to the typical viscoelastic MSD (plateau formation). The minimal gelling agent concentration is determined with the Time-Cure-Superposition (TCS) method. TCS consists in rescaling the MSD curves with factors a and b, which gives a characteristic v-shaped curve (Fig. 2-II) indicating the minimum gelling agent concentration (see application note "Gel point determination by TCS" for more details).

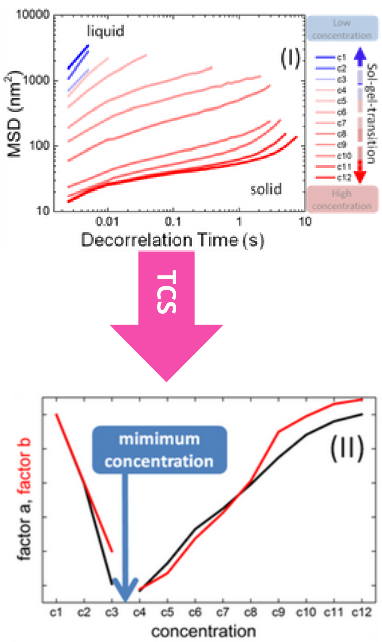

Figure 2: (I) MSD curves of different gelling agent concentrations. (II) TCS results: arrow indicates the minimal concentration to obtain a gel.

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B) What is the gel point for a given concentration?

As seen previously, the samples with the concentrations C4 to C12 form a gel: the gelling time for each concentration was determined by the TCS treatment as shown in Figure 2. The different gel points are listed in the formulation matrix at the bottom.

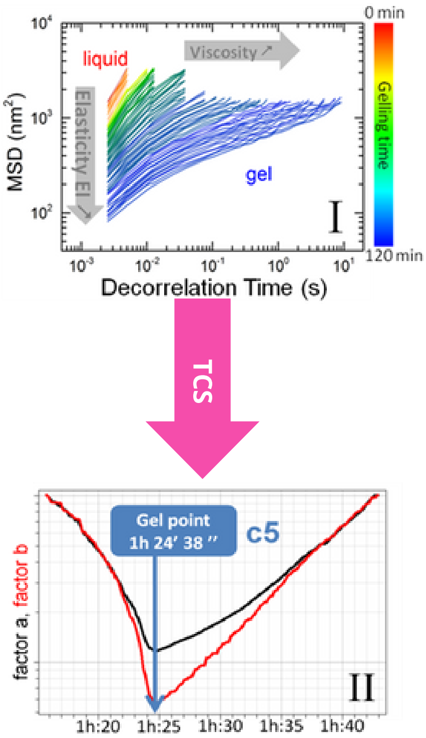

Figure 3: (I) Evolution of MSD during gelation. (II) Plot of factors a and b as a function of time for a given concentration: the characteristic v-shaped curve indicates the gel point.

C) What is the gel strength of each gel?

Rheolaser measures also the viscoelastic properties, such as the elasticity index (EI). The higher the EI, the higher the elasticity of the gel. Figure 4 shows the EI evolution of several concentrations (for better visibility only a few concentrations are plotted). The determined gel strength of each gel is listed in the formulation matrix below.

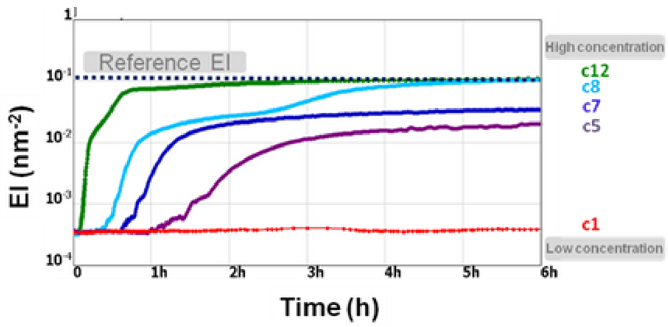

Figure 4. Influence of (a) formulation and (b) temperature on SLB to improve printability.

D) What time is needed to match a reference elasticity?

Rheolaser Master also helps to compare the different samples. Formulation's question might be how much time is needed to reach a reference elasticity, e.g. the elasticity of C12. The samples C8 to C11 also reach the reference elasticity, but with longer reaction times, as they are less concentrated.

Formulation Matrix

Thanks to this formulation matrix, the formulator can decide which is the optimal composition, depending on the formulation strategy. In our example, c8 is product-saving, whereas c12 is time-saving. The 12 concentrations studied here, were measured in two series of 6 samples with an operator time of less than 1 h.

Sample	c1	c2	c3	c4	c5	c6	c7	c8	c9	c10	c11	c12
a) Gel formation	NO	NO	NO	YES	YES	YES	YES	YES	YES	YES	YES	YES
b) Gel point	--	--	--	99'26''	84'38''	55'45''	45'21''	28'19''	21'14''	17'38''	13'44''	6'40''
c) Gel strength	--	--	--	0.015	0.020	0.028	0.034	0.11	0.11	0.12	0.13	0.13
d) Time to reach EI(ref)	--	--	--	--	--	--	--	4h46'	3h05'	2h36'	2h18'	2h08'

CONCLUSION

Rheolaser Master allows studying the gelation properties of unknown gelling agents. Due to its 6 measurement positions, the key parameters such as minimum gelling agent concentration, gelation time, and gel strength are determined rapidly and accurately.