Temperature dependent sol-gel reactions – Use of closed cell

GENERAL

Introduction

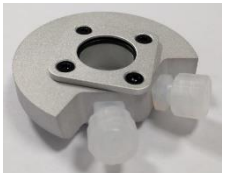
Biopolymer gel formation is most often temperature dependent. A good control of the temperature is one of the main criteria for studying such gels, as gelation point can be dependent on the ramp speed.This application note will show the use of the Rheolaser^{CRYSTAL} in temperature dependent gelation of carrageenan and gelatin. Rheolaser^{CRYSTAL} is equipped with a perfect temperature control between 4 and 90°C and controlled ramp speed between 0.1 to 25°C/min in heating and cooling mode. In addition, the measurement cell is perfectly hermetical avoiding evaporation.

KEY **BENEFITS**

- RAMP SPEED CONTROL
- LOW SAMPLE VOLUME
- NO EVAPORATION

Materials

Commercially available **κ-carrageenan and gelatin (160 and 280)** were completely dissolved at 90°C or 60°C. Carrageenan was used at different concentration and different NaCl content. 0.1wt% polystyrene particles were added prior to sampling in all polymer solution in order to ensure backscattering conditions.Sampling was done with a syringe to fill the closed cell bubble free with about 2 mL. The use of the closed cell is described in ANXXXX



Methods

Rheolaser^{CRYSTAL} is based on Diffusing Wave Spectroscopy (DWS), a multiple light scattering technique.
Light is scattered by the microstructures inside the sample (droplets, crystallites, etc.), creating an interference pattern called Speckle Image (figure 1). The variation of this image in time is directly related to the mobility of the microstructures; The faster they move, the faster the Speckle Image changes. By a mathematical analysis of this variation, a decorrelation functions can be computed and then processed to obtain the Microscopic Dynamic (Micro-Dynamics) as a function of temperature or time. Temperature ramps from 70°C to 20°C with 0.5°C/min, 1°C/min and 2°C/min were applied for the carrageenan samples, whereas gelatin samples were cooled down from 50°C to 4°C at 1°C/min, 3°C/min and 5°C/min.

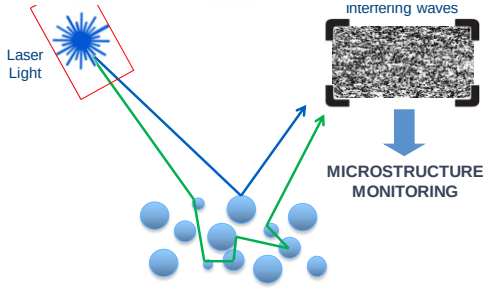


Figure 1: Experimental set-up of RHEOLASER Crystal.

Results and Discussion

a) Carrageenan

Figure 2 shows two cycles of cooling and heating of a 0.5wt% carrageenan dispersion with 20mM KCl at 1°C/min. The two full cycles overlap perfectly, showing that gel formation and dissolution are completely reversible, and the closed cell is perfectly hermetical.

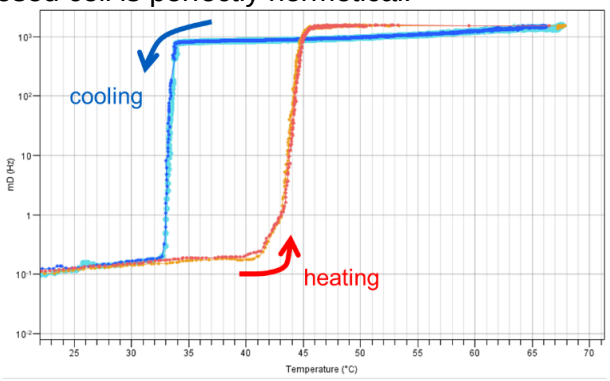


Figure 2: Micro-Dynamics of cooling and heating of a 0.5wt% carrageenan dispersion with 20mM KCl at 1°C/min. Two full cycles.

Figure 3 shows the heating and cooling of a 1 wt% carrageenan dispersion with 40mM KCl at two temperature ramps. Only slight differences are observed between 1°C/min and 2°C/min. For each temperature ramp, two full cycles are shown.

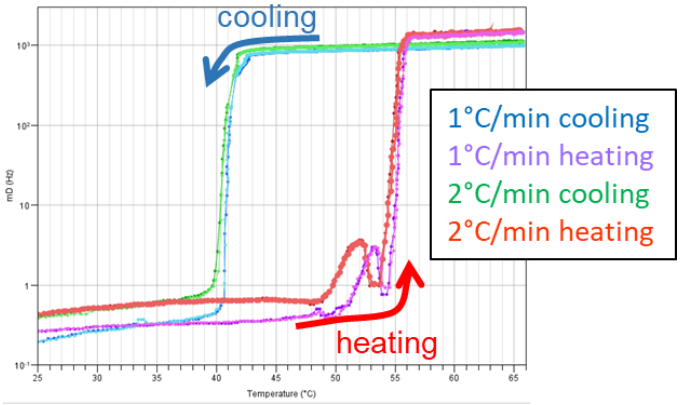


Figure 3: Micro-Dynamics of gelation and dissolution of carrageenan samples. Two full cycles.

b) Gelatin

Figure 4 shows gelation of a 9wt% gelatin dispersion at 1 and 5°C/min cooling ramp. Depending on the cooling ramp, the gelation point changed. By controlling the cooling ramp, the different gelation kinetics can be observed.

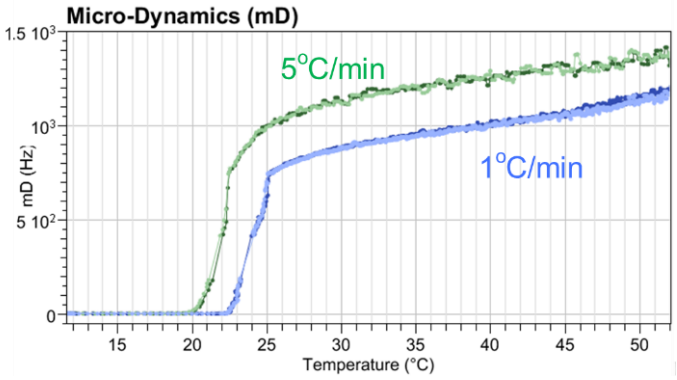


Figure 4: Gelation of 9wt% gelatin during cooling

Figure 5 shows the evolution of the gel point for different samples. Whereas gelatin samples show significant decrease of gel point temperature when we increase the cooling ramp, i.e. up to 2°C difference in gel point, the gel point of the carrageenan samples stayed equal (slight variation of 0.3°C, when increasing the cooling ramp).

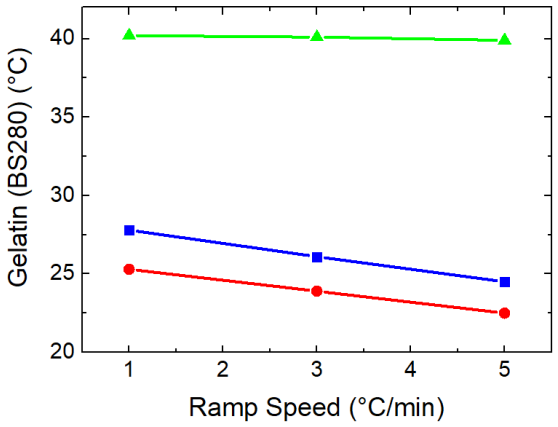


Figure 5: Gel points as a function of temperature ramps for different systems.

CONCLUSION

Rheoalser[CRYSTAL] allows to study temperature dependent sol-gel processes using the hermetically closed cell. The cooling ramp did not have major impact on gelation temperatures of carrageenan samples, but significant shift of gelation temperature of gelatin dispersions were observed when cooling ramp speed was changed. The faster the cooling, the lower the gel point.