RHEOLASER MASTER

Rheology of corrosive and hazardous materials

INTRODUCTION

Rheological studies of corrosives and hazardous compounds can be tricky. Degradation of the metal parts of the rheometers, as well as harm to the operator must be prevented. Rheolaser Master provides a clean and simple solution with disposable measurement cells. This protects the instrument and operator. This application note will show two typical examples with hazardous and reactive materials:

- Study of shear-thinning properties of corrosive materials.
- Determination of the minimal concentration of hazardous cross-linkers.

Operator protection | **Reactive materials** | **Corrosive materials**

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 1-left). The faster the Speckle Image changes in time, the higher the mobility of the microstructure.

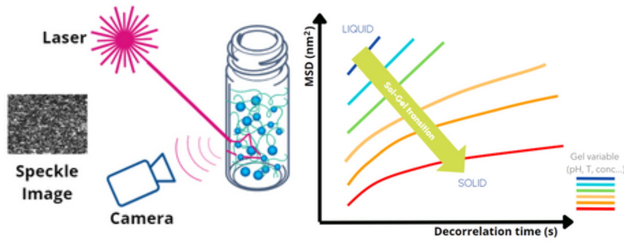

Figure 1. Schematical representation of the measurement set-up.

By mathematical treatment, Mean Square Displacement (MSD) curves are obtained (Figure 1-right), 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.

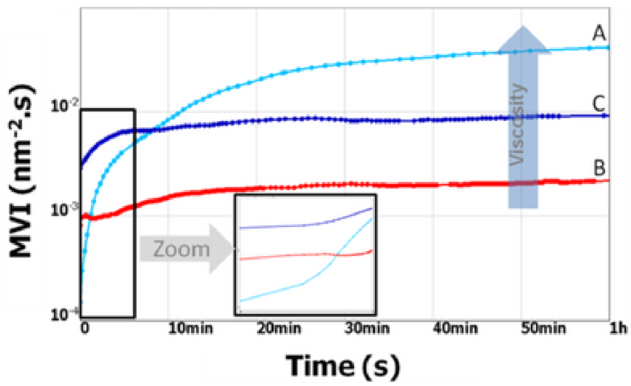

Figure 2. Evolution of MVI after stirring at 25°C.

Shear-thinning properties of corrosive materials

In the aerospace industry, weight reduction is the focus of airplane development. The use of lighter materials (polymer composites) and their adhesive bonding through chemical reactions instead of screws reduces fuel consumption. Fiber-reinforced Poly(Ether Ether Ketone) (PEEK/FC) has poor adhesion properties. The surfaces have therefore to be chemically activated, most often with a corrosive compound. Precise handling of the reactive formulation is difficult due to its very fluid character. Viscosity modifiers (polymers) introducing shear-thinning behavior are added to ensure proper handling: During spreading the formulation should have a low viscosity, whereas, at rest, the viscosity should increase rapidly to prevent sagging.

Due to the corrosive nature of the surface activator, metallic parts of conventional rheometers would be destroyed. Therefore, three mixtures with comparable activating efficiency, but different chemical compositions (see table below), were studied in disposable glass tubes with Rheolaser. The rheological tests analyzed the response of these mixtures to manual stirring and the subsequent recovery. Figure 2 shows the Microscopic Viscosity Index (MVI) just after stirring. The higher this index is, the higher the viscosity of the sample. Mixture A (high amount of polymer) is more shear thinning, becoming more fluid and less viscous after stirring (good spreading), and recovers fast to obtain very high viscosity (no sagging). This is in good agreement with the experiments performed in real conditions.

Table 1. Composition of mixtures for activators

	Quantity Activator	Quantity Polymer
Mixture A	Low ↓	High ↑ Polymer 1
Mixture B	High ↑	Low ↓ Polymer 1
Mixture C	Low ↓	High ↑ Polymer 2

This work was performed in collaboration with the research group SMODD (IMRCP, Toulouse) and AIRBUS INDUSTRIES.

Minimal concentration of a hazardous cross-linker

Building materials are most often cross-linked composite products. Rheological studies of gel curing are difficult due to the toxicity, volatility, and smell of some curing agents. In addition, the cleaning of the rheological geometries is almost impossible due to the low solubility of the cured gel. As a consequence, the handling of these gels is not comfortable. Rheolaser Master provides an easy alternative to study the curing using disposal glass tubes.

To reduce the amount of curing agent, one should know the minimum concentration to obtain a gel. For this issue, a polymer system is studied with different concentrations of curing agents. Rheolaser Master measures the particle Mean Square Displacement, which indicates the viscoelastic properties. Figure 4. I show the MSD curves of 9wt% gelatin solutions at 40°C with different cross-linker concentrations (glutaraldehyde). The MSD curves in the top left are those with a low concentration of curing agent. They are linear, which indicates liquid-like behavior. With increasing concentration, cross-linked gels are formed, leading to the typical viscoelastic curves (plateau formation). The minimum concentration is determined with the new Time-Cure-Superposition (TCS) method. TCS consists in rescaling the MSD curves with factors a and b, which gives a characteristic v-shaped curve (Figure 4. II) indicating the minimum curing agent concentration (see application note "Gel point determination by TCS").

Figure 3: Rheometer (left) and disposal measurement cell of Rheolaser Master (right).

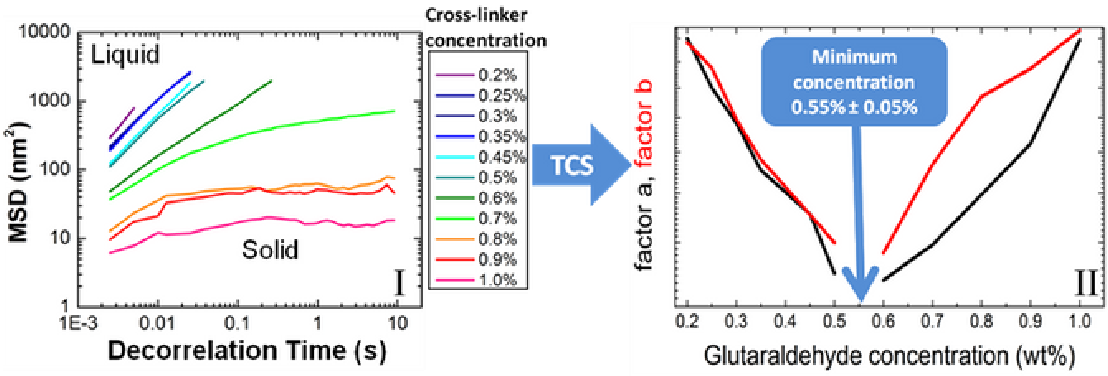

Figure 4. Mean Square Displacement of gelatin dispersions with increasing cross-linker concentrations.

CONCLUSION

Rheolaser Master uses disposable cells, which are hermetically closed. This is a useful help when dealing with corrosive and hazardous materials to protect both, the operator and the equipment. Thanks to the 6 simultaneous measurements operator time are reduced, minimizing the exposure.