AN035e

Systemic Rheology

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THE RHEO-REACTOR

Systemic rheology is the determination of rheological behavior under near real process conditions

INTRODUCTION

The elaboration of complex fluids whith special properties and functionality is based on formulation engineering, a multi-level science, encompassing process engineering, physico-chemistry, chemistry, rheology and interface sciences among others. Some objectives of formulation engineering are the quantitative analysis of the different steps of the manufacturing process, the mastering of those steps in order to relate to formulations established in the factory laboratory, the knowledge and monitoring of the rheology of the complex fluids in the course of its formulation.

An original tool integrating the knowlege of process engineering and rheology and which has been named “systemic rheology” has developped by Gemico in France /1/. To this effect, traditional geometries of the rheometers have been changed into stirred micro-reactors so that within certain steps of a process could be reproduced to some extent if not copied exactly. The test assembly constitutes the rheo-reactor operating in batch or semi-batch mode and which can perform mixing procedures, rheological monitoring in situ, i.e. without sampling and even rheological characterisations which are often difficult to realise with complex fluids. Some of these are actually extremely sensitive to their thermo-mechanical history and therefore cannot be tested in an application relevant manner outside of the process itself. Also complex materials may be of heterogeneous nature with a micro-structure of the same size as the gap of traditional geometries (cone-plate, parallel plates, Couette etc.). Conditioning and analysis of this kind of samples often meet with major experimental difficulties such as structural alterations of the material, slipping, sedimentation or characterization during an uncontrolled pre-strain state.

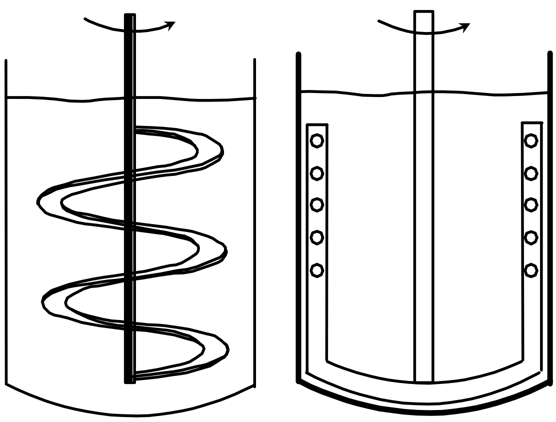

Figure 1. : Helicoidal ribon and ancer

The configuration of the rheo-reactor itself eliminates all these problems and at the same time it fulfills some of the objectives of formulation engineering /2/. The purpose of this paper is to show that it is possible to characterise a complex fluid from a rheological point of view within the configuration of the rheo-reactor.

METHOD

Working with the rheo-reactor is based on two steps: first, the traditional geometries are replaced by stirred micro-vases and, secondly, the assembly vase/stirring rod must be gauged to allow the

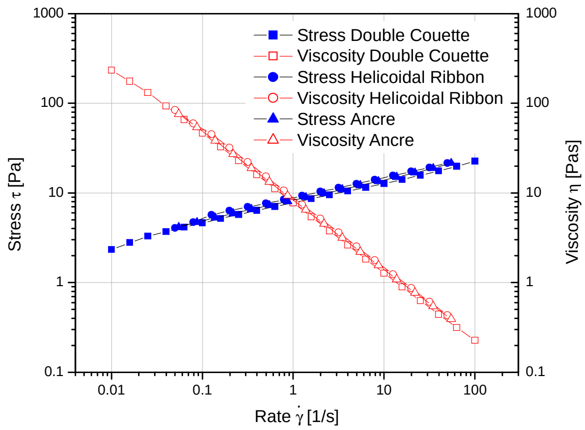

Figure 2.: Rate sweep

determination of the characteristic material functions of the respective material; the aim of the rheometer thus remains preserved. The first step is quite simply and consists of using a universal test fixture holder and replacing the test geometry by adapting a vase and stirring rod to the end of the axes (Fig. 1). This procedure can be performed on all rotational rheometers such as ARES, AR2000 and there predecessors RDAII, RFSII, AR1000.

As to the second step, advantage is taken from the option "special geometry" available in the [TA]Orchestrator software. Two constants were

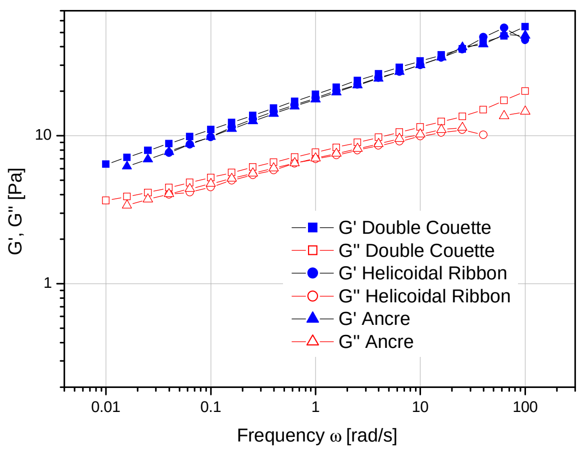

Figure 3. : Frequency sweep

introduced namely ($K\tau$), which relates the torque M and the stress τ and ($K\gamma$), relating the angular deformation θ and the deformation γ:

$$\tau = K\tau\ M \text{ and } \gamma = K\gamma\ \theta$$

These two constants can be determined by a gauging procedure allowing to extract the material functions from instrument data /3/ obtained with the special geometry. It consists in calculating the stresses and the deformations in a Couette, equivalent to a stirring system.

RESULTS

The graphs in fig. 2 to 4 show the compared test results measured in steady and oscillatory modes. The following geometries have been used:

- double wall Couette (DC), (∅ Bob = 47 mm, ∅ Cup = 50 mm),
- helocoidal ribbon (HR), (∅ Ribbon = 31 mm, step = 30 mm, ∅ Cup = 40 mm), and
- ancre (AN), (∅ Ancre 47 mm, ∅ Cup 50mm).

The sample used was a preparation for salad dressing (Maille). All measurements were con-

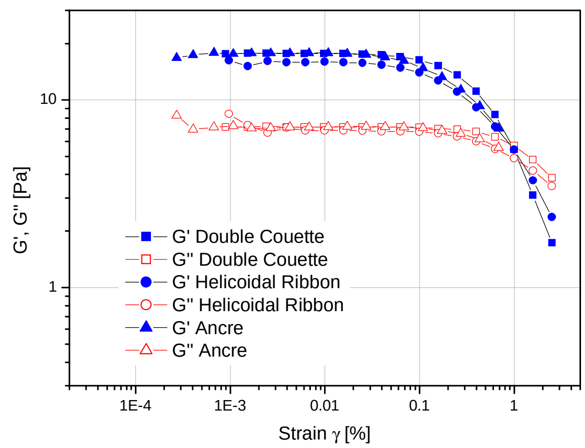

Figure 4.: Strain sweep

ducted at room temperature. The repeatability of the tests is comparable to that of measurments with conventional geometries.

Figure 2 shows the flow curves obtained from tests performed with the three geometries, DWC, HR, AC. The data obtained from the DWC cover a wider range than the data obtained from HR and AC. Good agreement between all three geometries exists after shifting the HR and AC data to superpose the DWC data.

Figure 3 shows dynamic frequency data. HR and AC deviate slightly at higher frequency. Complex flow behaviour around the the ribbon and the ancre as well as inertia effects are probably the reason forthese deviations.

Figure 4 shows a dynamic strain sweep. In the linear range, very good agreement can be seen between the three geometries. As expected from Figure 1, the sensitivity of the HR and the AC is slighly inferior to the Couette geometry at low strains. Minor differences can also be seen at the on-set on non-linear behavior. This is due to the difference in the flow pattern (simple flow in a Couette, complex flow around the ribbon and ancre), which causes the sample to behave differently. These differences, when more significant, can be the reason for the failure of using convential rheometer data to model real processes.

REFERENCES

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