

MENUSIM WORKSHOP
MEASURING THE RHEOLOGICAL PROPERTIES OF POLYMERIC MATERIALS

ATHENS, GREECE
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**Workshop
Report**

MENUSIM, a project sponsored by the EC to promote the exchange of information between universities and industries in the field of rheological MEasurements and NUmerical SIMulation for polymer processing purposes, organised a workshop which was held at the Hotel Fenix in Athens. This was the first MENUSIM workshop and a second which deals with the application of numerical methods in the field of polymer processing will be held from 25th to 28th October in Paris. The Athens meeting was dedicated to questions of measurement accuracy in rheometry, new methods and instruments as well as rheological properties of polymeric materials. 10 talks were presented and the schedule gave the participants plenty of time for discussion which was used extensively.

The first talk was given by Axel Göttfert (Göttfert Prüfmaschinen GmbH, Buchen, Germany, axel@goettfert.com) who presented *News from Capillary Rheometry*. During his very informative talk he analysed the state of the art in the field with special emphasis on the accuracy of pressure and temperature measurements. He raised the question how one can extend the coverable shear rate range, how the Bagley correction is performed and analysed correctly. Furthermore he informed about the determination of pressure dependent viscosities and the correct determination of extrudate swell.

The second talk *Characterization of a Calibration Polymer for Extrusion Purposes* was given by Claude Dehennau (Solvay, Brussels, Belgium) who is in charge of the preparation and the distribution of a polymer (HDPE) which can be used for calibration purposes in capillary rheometry. He presented the material (400 kg are now available) and the rheological measurements they performed. For this material the flow curve, the extensional viscosities and dynamic shear moduli are known in wide temperature, shear and extensional rate regions. Everybody who is interested to compare the own equipment with Solvay's rheometers is invited to contact Claude Dehennau (claude.dehennau@solvay.com) or the MENUSIM homepage: www.meca.ucl.ac.be/memawww/menusim/.

Philip Freakley, Loughborough University (UK, p.k.freakley@lboro.ac.uk), described *The Effects of Capillary Die Geometry and Wall Slip on the Extrudate Swell of Rubber Compounds* and

pointed out that, for such complex polymer materials, the state of mix is of tremendous importance, as it strongly influences the flow properties. He described a technique to assess wall slip of rubber compounds using a friction slider developed in his laboratory. During the discussion, he stressed that with such materials as rubber compounds, it is very difficult to ascertain that uncontrolled thermal effects (*i.e.* shear heating) do not spoil results.

Hartmut Braun from Haake GmbH (Karlsruhe, Germany, service@haake.de) analysed in his talk *Unmatched Accuracy of Dynamic Rheometry Data: Slogan or Reality* the errors appearing on the dynamic shear moduli measured with a controlled stress rheometer. This detailed analysis of all factors like inertia, bearing friction, break moments and others effecting the accuracy were presented and their relative importance for the overall error given. This talk introduced to the high complexity of rheometer dynamics that has to be considered for a complete error analysis and lights up the way to an even higher measuring standard. Furthermore, H. Braun showed that the targets of the corresponding ISO specification with respect to repeatability and reproducibility are not accomplished in everyday practice.

Henri Burhin, Alpha Technologies (UK, henri_burhin@alpha-technologies.com) gave a thorough description of *The Rubber Process Analyser* (RPA), a dynamic torsional tester especially designed to test very stiff materials such as filled rubber compounds. Essentially the test chamber is a biconical cavity working under pressurized condition (4 MPa) in order to offer easy sample loading with the capabilities to combine at will strain, frequency and temperature sweeps. He provided full explanation about the complex signal treatment that yields typical dynamic properties such as complex moduli or complex viscosities, with a typical reproducibility of 5 - 7%.

Jean Leopold Leblanc, UPMC-LRMOP (Paris, jleblanc@ccr.jussieu.fr) gave two contributions: One on *A Prototype Sliding Cylinder Rheometer for the Study of Complex Materials at Very Low shear rates*. The use of the SCR allows the determination of shear viscosity in the very low shear rate region (typically < 0.0001 1/s), that is below the capabilities of rotational rheometers. The second talk was dedicated to the presentation of *Special*

testing techniques for filled rubber compounds with a torsional dynamic tester (i.e the RPA, see H. Buhrin's contribution). With the SCR, he demonstrated how the sample anisotropy can significantly affect the measured shear viscosity. A paper describing this research device will appear soon in the journal Polymer Testing.

A second talk about a calibration polymer was given by Konrad Fuchs (kfuchs@fmf.uni-freiburg.de) from Freiburg University: *Characterization of a Calibration Polymer for Rotational Rheometers*. A polystyrene of very narrow molecular weight distribution was chosen as the calibration material for rotational instruments. K. Fuchs presented the molecular data of this polymer and the corresponding dynamic shear moduli measured on different rotational, stress and strain controlled instruments. On this basis he was able to discuss the overall accuracy of moduli measurement which is in agreement with the results presented by Braun. Everybody who is interested in receiving a sample of this polymer should contact the University of Freiburg (chf@fmf.uni-freiburg.de) or visit the above mentioned MENUSIM web site.

Another interesting talk was given by Manfred Wilhelm from Max-Planck-Institut für Polymerforschung (Mainz, Germany, wilhelm@mpip-mainz.mpg.de) who presented a method allowing the analysis of non-linear viscoelastic properties of materials: *Fourier Transform Rheology and Non-Linear Properties of Polymers*. This methodology developed by himself and his co-workers allows the determination of higher harmonics appearing in the output signal of an oscillatory experiment with large deformation amplitudes. All this is done with extremely high accuracy and in a short period of time. M. Wilhelm presented results for very different polymeric systems. Due to the fact that non-linear properties are highly dependent on topology of polymers or morphology of heterogeneous polymeric systems this method might become an efficient way to explore these features of the mentioned materials.

Finally, Dario Nichetti from Pirelli Pneumatici, Italy (dario.nichetti@pirelli.com) presented results on *The Effect of Silanisation Level on the Rheology of Uncured Silica Compounds*. Experiments have been conducted in order to define a relationship between level of silanisation of sili-

ca, viscoelastic properties of the compounds and their processing conditions. The authors have shown that material properties are strictly related to the level of silanisation obtained during the mixing process. Moreover, this correlation can be also used to define the "best process condition".

In general, this workshop has shown the versatility and future perspectives of rheological techniques, but also the demand for further improvement of accuracy standards in rheological measurements. In this respect, the presented calibration fluids might play an important role.

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