Report Name: App Note 004-OLR

Comparison with a Controlled-Strain Rheometer

Here we demonstrate that the Rheology Solutions OnLine Rheometer (OLR) correlates well with the results measured by a conventional rheometer.

A genuine process monitoring and control rheometer which can be operated in-line or on-line and measure the viscoelastic properties in a flow environment and in real-time has the potential to improve product quality and reduce processing costs.

Standard reference material SRM2490 as labelled by the National Institute of Standards and Technology (NIST) was measured by the OLR and compared to rheological data measured by NIST using a controlled strain rheometer.

ONLINE RHEOMETER

To date, the control of the properties of process flows has been attempted by the use of capillary viscometers, vibrating probe viscometers, and slit rheometers. In-line process viscometers by their very nature yield only viscosity data, and hence provide an incomplete analysis of viscoelastic fluids. All of the instruments mentioned above operate only at a single shear rate (or frequency), thereby potentially limiting the usefulness of the data.

If more detailed knowledge of the viscoelastic properties of the fluid is required to control the properties, quality, and reproducibility of the product, test volumes must be removed from the process stream and measured off-line using an instrument such as a controlled strain rheometer. However, this is a time consuming procedure that may require the process line to be shut down until the results are available to confirm, or otherwise, the quality of the product. Alternatively, the process flow may continue with the potential loss of product. In some cases, the volume of lost fluid could be large and this could be particularly problematic when processing high-value materials (such as pharmaceuticals) or products that cannot be reprocessed or products that incur a disposal cost (for example, an environmental levy).

Thus a genuine process monitoring and control rheometer which can be operated in-line or on-line and measure the viscoelastic properties in a flow environment and in real time has the potential to improve product quality and reduce processing costs. The OLR is based on the squeeze flow method which is quite different from the controlled-strain instruments commonly employed off-line for quality control.

Before an instrument based on a new technology can be introduced into an industrial environment, it is necessary to ensure that the results obtained can be correlated with those obtained from the commonly used off-line instruments.

COMPARISON WITH CONTROLLED-STRAIN RHEOMETER

Standard reference material SRM2490 from the National Institute of Standards and Technology (NIST) was measured by the process control rheometer and compared to rheological data measured by NIST using a controlled-strain rheometer.

The modified Cox-Merz rule was applied to the rheological data as measured by the OLR and a controlled strain rheometer resulting in a very good correlation between the two sets of data.

MODIFIED COX-MERZ RULE

The modified Cox-Merz rule is an empirical relationship between the shear rate dependence of the steady-shear viscosity η, and the frequency dependence of the modulus of complex viscosity η^*, at equal values of correct angular frequency $k\omega$, and shear rate $\dot{\gamma}$ as given by Equation 1.

$$|(\eta^* (k\omega))| = \eta(\dot{\gamma}) \quad (1)$$

This relationship allows the rheological properties of the OLR (which applies an angular frequency ω) to be compared to the rheological properties of a controlled-strain rheometer (which applies a shear rate $\dot{\gamma}$).

By applying a shift factor k = 0.625 a very good correlation was found between the two data sets as shown by the graph of complex viscosity as a function of shear rate shown right.

This conclusion suggests that results obtained with the OLR in an in-line or on-line application could be used in place of those from an offline controlled- strain rheometer for process and quality control. Clearly the correlation between the two sets of data would have to be established for each fluid application, as a different shift factor is expected for different materials. The exact correlation would of course depend on the fluid under test and might well be less simple than the modified Cox-Merz rule used here. Using a shift factor, analogous to the Cox-Merz rule for viscosity, the storage modulus (elastic component) and the loss modulus (viscous component) measured using the OLR squeeze flow method show a very good correlation with the controlled-strain measurements reported by NIST. The ability of the OLR to measure the viscoelastic properties of a fluid over a range of frequencies in a very short time makes it an ideal instrument for real time process control.

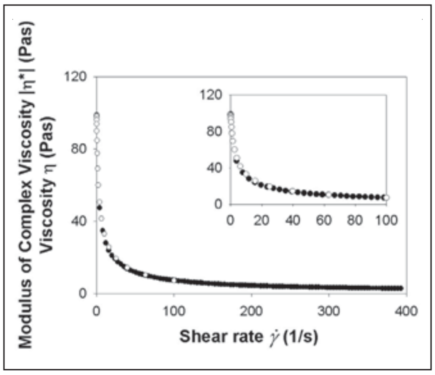

Figure 1 Viscosity as a function of shear rate of SRM2490 where the symbol (•) represents data measured using the OLR and symbol (◦) data measured using a controlled-strain rheometer.

Rheology Solutions

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