Online Process Rheometry Using Oscillatory Squeeze Flow

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Abstract:
The flow of complex fluids is routinely encountered in a variety of industrial manufacturing operations. Some of these operations use rheological methods for process and quality control. In a typical process operation small quantities of the process fluid are intermittently sampled for rheological measurements and the efficiency of the process or the quality of the product is determined based on the outcomes of these measurements. The large number of sample-handling steps involved in this approach cost time and cause inconsistencies that lead to significant variability in the measurements. These complications often make effective process/ quality control using standard rheometric techniques difficult. The effectiveness of control strategies involving rheological measurements can be improved if measurements are made online during processing and sampling-steps are eliminated. Unfortunately, online instruments capable of providing sufficiently detailed rheological characterisation of process fluids have been difficult to develop. Commercially available online instruments typically provide a single measurement of viscosity at a fixed deformation rate. This dependence on a single pre-determined shear rate restricts these instruments from identifying changes in the product or the process, especially if the viscosity at the pre-determined shear rate remains unaltered during these changes. We introduce an Online Rheometer (OLR) that uses small amplitude oscillatory squeeze flow to measure the viscoelastic properties of process fluids in-process and in real time under typical processing conditions. We demonstrate that with an appropriate measuring geometry and amplitude of oscillation, the frequency response of typical non-Newtonian fluids can be accurately measured in a process pipe. We also compare our results with other techniques that are typically used for process rheometry, critically evaluating the utility of the OLR technology for advanced process and quality control.

Zusammenfassung:
Das Fließverhalten komplexer Fluide wird gewöhnlich in einer Vielzahl von industriellen Verarbeitungsprozessen wiedergefunden. Bei einigen Verfahren werden rheologische Methoden zur Prozess- und Qualitätskontrolle angewandt. Bei einem typischen Verfahren werden geringe Mengen des Prozessfluids für rheologische Untersuchungen entnommen. Dann wird die Wirksamkeit des Prozesses bzw. die Produktqualität basierend auf den Ergebnissen dieser Messungen bestimmt. Die Vielzahl dieser Testschritte bei diesen Verfahren kostet Zeit und verursacht Inkonsistenzen, die zur signifikanten Schwankung bei diesen Messungen führen. Diese Komplikationen machen oftmals eine effektive Prozess-/Qualitätskontrolle mittels standardisierter rheometrischer Messungen schwierig. Die Effizienz der Kontrollstrategien mit rheologischen Untersuchungen kann verbessert werden, wenn Messungen online während der Verarbeitung durchgeführt werden und dabei die Probenentnahme fortfällt. Jedoch ist die Entwicklung von online-Messgeräten schwierig, die eine ausreichende rheologische Charakterisierung des Prozessfluids durchführen können. Kommerziell erhältliche online-Instrumente führen typischerweise eine Einzelpunktmessung der Viskosität bei einer bestimmten Deformationsrate durch. Die Abhängigkeit von einer einzelnen vorgegebenen Scherrate schränkt diese Messgeräte für die Identifikation von Produkt- oder Prozessänderungen ein, insbesondere dann, falls sich die Viskosität bei der vorgegebenen Scherrate durch diese Schwankungen nicht ändert. Wir stellen ein online-Rheometer (OLR) vor, dass eine oszillatorische Quetschströmung mit kleiner Amplitude ausnutzt, um die viskoelastischen Eigenschaften des Prozessfluids während des Prozesses und in Realzeit unter typischen Prozessbedingungen zu messen. Wir zeigen, dass mit einer adäquaten Messgeometrie und Oszillationsamplitude die Frequenzantwort eines typischen nicht-Newtonschen Fluids in einem Prozess mit einer Rohrströmung genau ermittelt werden kann. Darüber hinaus vergleichen wir unsere Resultate mit denen von anderen Techniken, die typischerweise für online-Rheometrie verwendet werden. Dabei wird der Nutzen der OLR-Technologie für eine anspruchsvolle Prozess- und Qualitätskontrolle kritisch evaluiert.

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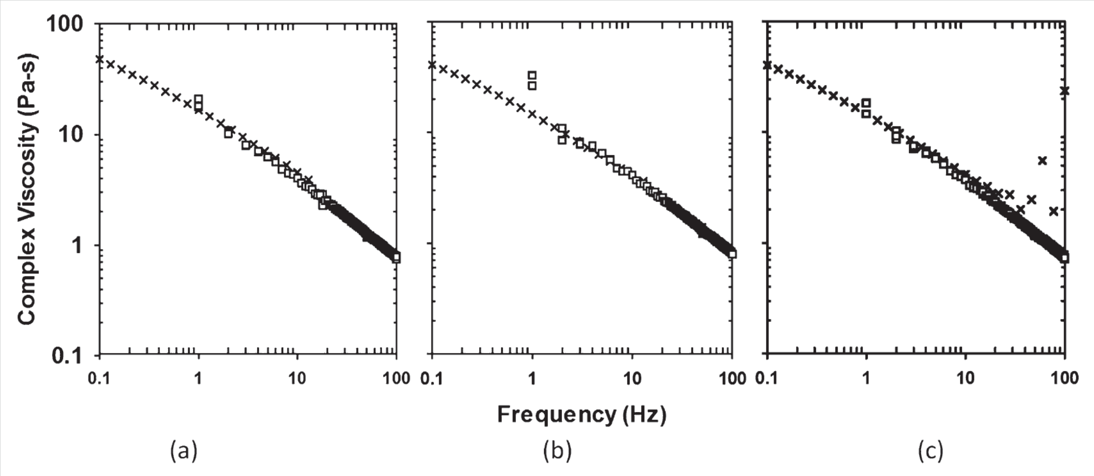

Figure 6: Viscosity measurements of 2.5 % solution of carboxymethylcellulose in water measured at various flow rates in the pipe loop. For comparison, measurements on a laboratory rotational rheometer are included using cross symbols (X). The other symbols represent measurements made by the OLR for repeated experiments at a fixed flow-rate. Results from three representative flow rates are shown: (a) $\dot{m}$ = 1500 kgs/hr (v ~ 0.1 m/s), (b) $\dot{m}$ = 1900 kgs/hr (v ~ 0.13 m/s) and (c) $\dot{m}$ = 2900kgs/hr (v ~ 0.2 m/s).

adopt technologies that can be used to design, analyse and control manufacturing through timely measurements of critical quality and performance attributes of raw and in-process materials with the goal of ensuring final product quality. These "Process Analytical Technologies" or PAT are expected to improve product quality, reduce waste and improve the productivity of lean manufacturing operations. Following the release of the roadmap industry practitioners have included a wide variety of instruments that enable them to monitor and control processes better. We believe that the OLR can contribute to this effort. In a review of the available instruments for process viscometry completed over a decade ago Zimmer et al observed that the process viscometers provided "grossly inaccurate" results when non-Newtonian fluids were of interest and suggested methodologies for correcting for the inaccuracies. Unfortunately the performance of process viscometers has not improved much since that appraisal. In this paper we have presented the performance of an OnLine-Rheometer (OLR) that provides accurate data for a range of non- Newtonian fluids and which provides measurements that agree quantitatively with measurements made on research-grade laboratory instruments. We have defined the flow configurations in which the instruments are likely to be used and have provided representative measurements for each scenario. In all cases a single calibration of the instrument is sufficient to provide effective and accurate characterisation of the process fluid in on-line and in real time.

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