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# Towards online, continuous monitoring for rheometry of complex fluids



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#### ABSTRACT

This paper presents an overview of the developments that have been made towards the design of an inline rheometer that has the capabilities for monitoring in real time the viscous constitutive parameters of non-Newtonian fluids in a pipe flow. This has potential applications for a wide range of fluids, including hydrocolloid solutions and polymer solutions. This is of relevance to many industries, for example the pharmaceutical, lubrication, food and printing industries. The use of mathematical algorithms for inferring rheological parameters from properties of flow field statistics is explored. Particular focus is given to the development of a flow cell rheometer containing a T-junction geometry with the capacity to induce a range of shear rates in the vicinity of the bend, and a distribution of elongational viscosities along the back-wall. Such features create an information-rich flow field that is beneficial for the development of a rheometer with a fast response time that is suitable for commercial purposes.

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#### 1. Introduction

The processing of complex fluids occurs in many engineering applications and in biomedical analysis. Efficiency of such processes can be crucially dependent on the rheology of the fluid. Viscous

the monitoring of viscous parameters requires samples to be taken manually and then analyzed in a cone-and-plate rheometer, or similar device. The ability to perform online continuous rheological characterization in real-time would permit tighter monitoring and control in a wide range of applications that involve the processing of fluids of non-Newtonian fluids, such as those in the food and beverage [1],

pharmaceutical [2], oil and lubrication [3], and high-tech printing

parameters can affect the texture or appearance of a fluid, and also they can determine the shelf-life stability of a product. At present,

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industries [4]. This would lead to a reduction in wastage caused by processes going off spec, and in the level of manual intervention required for product sampling. Currently, no such commercially available device exists that can perform this task efficiently for a broad range of fluids. Quality control is an endpoint test which results in disposal of below spec product. Continuous monitoring would reduce this wastage, potentially completely. Generally the wastage level of a wide range of processes is around 5%. Recently, much attention has been given to the use of flow configurations that yield an information rich data set of measurable flow parameters that can be mapped, through the solution of an inverse problem, to individual constitutive parameters. With the pressure to develop sustainable materials (biomass derived or utilizing waste products) for processing and manufacturing, industry must respond to the greater variability in input feedstock specification [5], as well as material properties varying during processing, with better control systems. Hence there currently exists a strong driver for online, continuous rheometry.

The rheological behavior of different types of non-Newtonian fluids is described by different constitutive equations. Some examples are discussed below.

#### 1.1. Power law fluids

The viscosity of power law fluids is given by

$$\mu = K\dot{\gamma}^{n-1},\tag{1}$$

where K is the flow consistency index (SI units Pa s<sup>n</sup>),  $\dot{\gamma}$  is the shear rate (SI unit s<sup>-1</sup>), and n < 1 is the flow behavior index. K and n denote the constitutive parameters of the fluid in question and it may be desirable to know what they are in any given situation [6]. For example, the viscosity of bio-oils is highly temperature dependent and follows a power law constitutive equation. The Krohne viscoline inline viscometer manufactured by Delta Instrumentation (http:// www.deltainstrumentation.com/viscosity/viscometer.html) is a novel device that can be used to determine the two rheological parameters, K and n, for power law fluids. The viscoline comprises a section of pipeline that can be inserted into a fluidic process. The pipeline contains two low pressure drop static mixers that are used to generate two different shear regimes. If the flow rate is known then the pipe line viscosity can be calculated from the two pressure drop measurements. The viscoline can operate at shear rates in the range 50 to  $500 \text{ s}^{-1}$ and at temperatures of -5 to 200 °C.

#### 1.2. Carreau fluids

For Carreau fluids, models with either three or four parameters are in common usage [7]. In the three parameter version, the viscosity at infinite shear rate is taken to be zero, and the viscosity is given by

$$\mu = \mu_0 \Big( 1 + (\lambda \dot{\gamma})^2 \Big)^{(n-1)/2} \tag{2}$$

where  $\mu_0$  is the zero-shear-rate viscosity,  $\lambda$  is a parameter with dimensions of time and n is the power-law exponent.

The four parameter Carreau model takes the form

$$\mu = \mu_{\infty} + (\mu_0 - \mu_{\infty}) \left[ 1 + \left( \lambda (\dot{\gamma})^2 \right)^{(n-1)/2}, \right]$$
 (3)

where  $\mu_{\infty}$  is the viscosity at infinite shear rate.

#### 1.3. Viscoelastic fluids

Viscoelastic materials possess both viscous and elastic characteristics when undergoing strain, and therefore exhibit time dependent

stress. The Phan–Thein–Tanner (PTT) constitutive law [8] uses the following form for the non-dimensional constitutive equation:

$$\begin{split} \textit{We}\tau_t &= 2\mu_1 \mathbf{D} - h\tau \\ &+ \textit{We} \Big\{ \boldsymbol{\tau} \cdot \nabla \mathbf{U} + (\nabla \mathbf{U})^T \cdot \boldsymbol{\tau} - \mathbf{U} \cdot \nabla \boldsymbol{\tau} + \boldsymbol{\zeta} \Big[ \mathbf{D} \cdot \nabla \boldsymbol{\tau} + (\mathbf{D} \cdot \boldsymbol{\tau})^T \Big] \Big\}, \end{split} \tag{4}$$

where *h* is usually defined using one of the following three forms:

Linear model : 
$$h = 1 + \frac{\epsilon We}{\mu_1} \operatorname{trace}(\tau),$$
 (5)

Quadratic model : 
$$h = 1 + \frac{\epsilon We}{\mu_1} \operatorname{trace}(\tau) + \frac{1}{2} \left[ \frac{\epsilon We}{\mu_1} \operatorname{trace}(\tau) \right]^2$$
, (6)

Exponential model : 
$$h = \exp\left[\frac{\epsilon We}{\mu_1} \operatorname{trace}(\tau)\right],$$
 (7)

where

- $\tau = \mathbf{T} 2\mu_2 \mathbf{D}$ ,
- T is the extra-stress tensor,
- **D** is the rate of deformation tensor,
- **U** is the velocity vector,
- $\mu_1$  is the polymeric viscosity,
- $\mu_2$  is the solvent viscosity,
- We is a Weissenberg number defined as  $We = \left[\frac{U\lambda}{L}\right]$ , where  $\lambda$  is a relaxation time and U and L are typical velocity and length scales respectively,
- $\epsilon$  and  $\zeta$  are positive constants used to control the viscoelastic properties.

The aim of this article is to provide an updated review, with a particular focus on industrial aspects that would potentially benefit from improvements in the ability to perform online rheometric characterization in real-time. Applications of rheometry in engineering, manufacturing and medical processes are outlined in Section 2. Capillary methods for rheological characterization are described in Section 3. In Section 4 some recent research that uses advanced mathematical algorithms to solve inverse problems in order to determine rheological parameters in real time, is outlined. Discussion on the effect of increased variability of feedstocks as a key driver for the development of improved devices for determining rheological parameters online forms the theme for Section 5, where the present situation is summarized.

#### 2. Applications for rheometry

#### 2.1. Engineering monitoring and control

The monitoring and control of rheological parameters are of importance for a range of engineering and processing systems. Some examples are discussed in this section.

Melt flow index (MFI) as a single data point experiment is a well-established technique for measuring changes in the average molecular weight of a polymer melt, although its accuracy is not always reliable as it refers to only a single point measurement within a larger spectrum of properties [9]. MFI is a measure of the ease of flow (pliability) of a thermoplastic polymer melt. It is defined as the mass in grams of a polymer passing through a standardized capillary of 2 mm diameter and 8 mm length over a 10 min interval, at a specified temperature, when a standardized load is applied. The MFI only tests a material at a single shear stress and temperature. It provides an indirect measure of molecular weight. A higher MFI is indicative of a lower material viscosity. Many processes use MFI to assess the viscous properties of incoming materials and as an aid to monitoring changes in the process. More accurate rheology tests can be performed using capillary or parallel plate rheometers.

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