TRANSIENT MEASUREMENTS IN RATE CONTROLLED RHEOMETERS: A NEW METHOD OF STANDARDIZATION

IMANE TALBI-BOUCENNA AND DANIEL QUEMADA

Laboratoire de Biorhéologie et d'Hydrodynamique Physico-chimique, CNRS ESA 7057
2, Place Jussieu, case 7056, 75005 Paris, France
Fax: x33.1.44274335
e-mail: bouccen@ccr.jussieu.fr and quemada@ccr.jussieu.fr

ABSTRACT:
Rheological measurements in response to transient shear rate exhibit coupling effects between mechanical characteristics of rheometer and unsteady (thixotropic and viscoelastic) properties of fluid. This coupling is observed if the characteristic times of the latter lie in the range of those associated to mechanical properties of the apparatus. Damped oscillations observed in the stress relaxation curves obtained with some standard oils submitted to stress steps are an example of this type of anomalous responses. This work introduces a new method of standardization for a low shear rate rheometer (Contraves LS40) under unsteady flow conditions. This "standardization under transient conditions" is developed in order to obtain a correct interpretation of unsteady measurements performed on elastic and thixotropic fluids, hence to permit modelling of the latter. A second order differential operator is supposed capable to describe the fluid-rheometer coupling. A series of measurements on standard silicone oils, which include electromechanical characteristics of the instrument, allow us to determine the parameters involved in this operator. Then, a preliminary test of this method is performed on a typical system (Red Blood Cell suspension with Dextran) which presents viscoelastic and thixotropic behavior.

ZUSAMMENFASSUNG:
Die rheologischen Antwortfunktionen bei einem Spannversuch zeigen Kopplungseffekte zwischen den mechanischen Charakteristika des Rheometers und den instationären (thixotropen und viskoelastischen) Eigenschaften des untersuchten Fluids. Diese Kopplung wird dann beobachtet, wenn die charakteristischen Zeitskalen der instationären Fluideigenschaften im Bereich der charakteristischen Zeitskalen der mechanischen Eigenschaften des Messapparates liegen. Die gedämpften Oszillationen, welche in den Spannungsrelaxationskurven von Standardölen unter Spannversuchen beobachtet wurden, sind ein Beispiel dieser anomalen Antwortfunktion. Diese Arbeit stellt eine neue Methode zur Standardisierung für ein schubspannungskontrolliertes Rheometer (Contraves LS40) unter instationären Fliessbedingungen vor. Diese Standardisierung bei instationären Fliessbedingungen ermöglicht eine korrekte Interpretation von instationären Messungen an elastischen und thixotropen Fluiden und somit die Modellierung dieser Flüssigkeiten. Es wird angenommen, dass sich die Fluid-Rheometer Kopplung mit einem Differentialoperator zweiter Ordnung beschreiben lässt. Mit einer Reihe von Standardsilikonölen, welche auf die elektromechanische Eigenschaften des Rheometers abgestimmt sind, werden die Parameter in diesem Operator bestimmt. Im Anschluss wird dieser Methode an einem typischen System (Suspension roter Blutkörperchen mit Dextran), welches viskoelastisches und thixotropes Verhalten zeigt, getestet.

RÉSUMÉ:
Les mesures rhéologiques en réponse à des échelons de cisaillement montrent l'effet de couplage entre les caractéristiques mécaniques du rhéomètre et les propriétés instationnaires (thixotropiques et viscoélastiques) du fluide. On peut observer ce couplage si les temps caractéristiques du fluide se situent dans le domaine de ceux associés aux propriétés mécaniques de l'appareillage. Les oscillations amorties que montrent les mesures à cisaillement imposé sur des huiles étalon sont un exemple de ce type de réponses. La présente étude concerne le développement d'une méthode d'étalonnage en régime d'écoulement instationnaire applicable à un rhéomètre à cisaillement imposé (Contraves LS40). Le but de cet "étalonnage dynamique" est de corriger les mesures instationnaires obtenues pour des fluides élastiques et thixotropes et ainsi d'en permettre la modélisation. Un opérateur différentiel du second ordre est supposé décrire le couplage fluide-rhéomètre. Une série de mesures effectuées sur des huiles étalons de silicone ont mis en évidence les caractéristiques électromécaniques du rhéomètre et ont permis de déterminer les paramètres de l'opérateur. Des essais préliminaires de cette méthode ont été effectués sur un système présentant un comportement viscoélastique et thixotrope (suspensions de globules rouges en présence de Dextranes).

KEY WORDS: Transient flow, inertial effects, time-dependent properties, stress relaxation

© Appl. Rheol. 10, 6, 280-287 (2000)

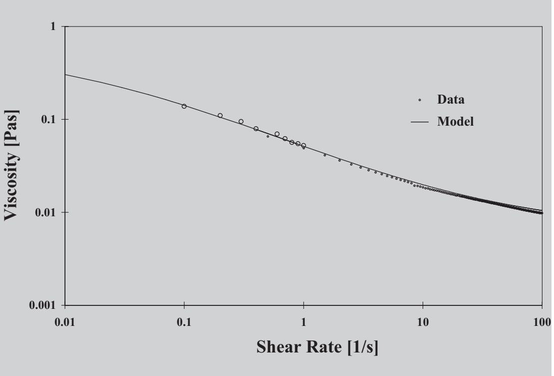

Figure 10 (left): Steady viscosity versus shear rate and model curve calculated with Eq. 5 for suspension of washed Red Blood Cells in 3% Dextran solution (η_0 = 633mPas, η_∞ = 6 mPas, $\dot{\gamma}_c = 9.5\ s^{-1}$, p = 0.43).

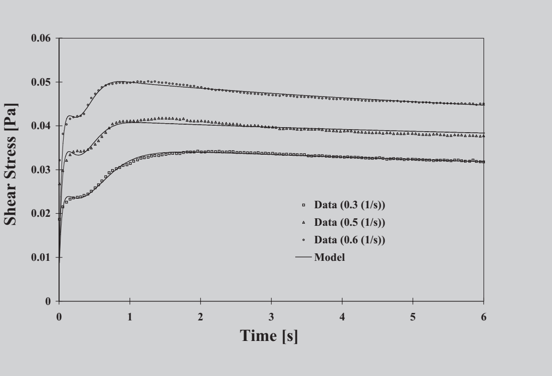

Figure 11 (right): Shear stress versus time and model curves calculated with Eq. 6 for suspension of washed Red Blood Cells in 3% Dextran solution (s_1 = 3;

$\dot{\gamma} = 0.3 s^{-1}$: $\kappa_A = 1.5\ s^{-1}$; G = 8 mPa; t_R = 4.2 s;

$\dot{\gamma} = 0.5 s^{-1}$: $\kappa_A = 4.3\ s^{-1}$; G = 8 mPa; t_R = 4.9 s;

$\dot{\gamma} = 0.6 s^{-1}$: $\kappa_A = 6.5\ s^{-1}$; G = 8 mPa; t_R = 6.7 s).

This procedure leads to determine the elastic modulus, G, the mean relaxation time characterizing the aggregate formation, $t_A = \kappa_A^{-1}$, and the retardation time, t_R. In order to test the method we discuss next the results of red blood cell suspensions. These measurements concern suspension of washed red blood cells in salt solution with addition of Dextran (molecular weight M_W = 72200, concentration c = 3%) where the presence of aggregation is due to this Dextran addition and leads to thixotropic behavior. The measurements were performed at constant hematocrit (= 45%) close to the RBC volume fraction and constant temperature (20°C). The modeling of the thixo-elastic behavior requires the knowledge of the steady parameters, η_0, η_∞, p, and $\dot{\gamma}_c$, that are directly obtained by flow measurements (see Fig. 10) and the unsteady variables κ_A (or κ_D), G and t_R that are the only free parameters. Results of the data fitting are represented in Fig. 11 and show a very good agreement between the model and the experimental data. The variation of the unsteady parameters for different shear rates and different levels of thixotropy will be discussed in a forthcoming publication.

6 CONCLUSION AND PERSPECTIVES

In the case of shear rate controlled rotating rheometers a phenomenological differential equation was used to describe the bob's movement. A "standardization under transient conditions" for transient shear stress measurements, which includes the main (electromechanical) part of the fluid-bob coupling is determined from a series of measurements on standard silicone oils. In spite of its approximate character the proposed method gives satisfactory predictions of the transient responses of the shear rate controlled rheometer. In fact, the method consists in determining the transfer function of the apparatus described by a second order differential operator equivalent to that involved in the pure mechanical case, but with coefficients which have an electromechanical origin directly related to the measuring head characteristics. The validity of the proposed method has been checked on standard oils in order to confirm the possibility of using this operator for analyzing, at least approximately, the stress response in the presence of inertial effects. It has been shown that the method could be extended to rheological characterization of non-Newtonian ("thixo-elastic") fluids. Examples of predictions of shear stress versus time variation using a thixo-elastic model taking this standardization into account have been satisfactorily compared to three experimental data on washed red blood cell suspensions in presence of thixotropy. Data modeling of RBC suspensions for different shear rates and different levels of thixotropy will be discussed in a forthcoming publication.

ACKNOWLEDGMENTS

The authors thank P. Flaud for helpful discussions.

REFERENCES AND FOOT NOTE

[1] I. M. Krieger: J. Rheol 34 (1990) 471-483.
[2] C. Baravian, D. Quemada, A. Parker: J. Texture Studies 27(1996) 371-390.
[3] C. Baravian, D. Quemada: Les Cahiers de Rhéologie 14 (1996) 653-668.
[4] C. Baravian, D. Quemada: Rheol. Acta 37 (1998) 223-233.
[5] J. Passard, R. Kouitat Njiwa, P. Perré: Eur. Phys. J. AP 3 (1998) 321-342.
[6] Operating Instructions: Low Shear 40, Technical report, Mettler - Toledo AG (1991)
[7] J.C. Healy and M. Joly: Biorheology 12 (1975) 335-340.
[8] D.C. Cheng, F. Evans: Brit. J. Appl. Phys. 16 (1965) 1599.
[9] D. Quemada: Biorheology 30 (1993) 253-265.
[10] D. Quemada: Eur. Phys. J. AP 1 (1998) 119-127.
[11] D. Quemada:Eur. Phys. J. AP 5 (1999) 191-207.

APPENDIX A: NUMERICAL INTEGRATION OF THE EQUATION OF MOTION

Numerical resolution of the equation of motion $\alpha \frac{d^2\theta}{dt^2} + D\frac{d\theta}{dt} + E\theta = 0$ allows us to obtain the variable θ(t). Knowing that

This is an extract of the complete reprint-pdf, available at the Applied Rheology website
http://www.appliedrheology.org

Table 2: Values of maximum torque measured for each sensitifity.

Sensitivity	1	2	3	4	5
Maximum torque (μNm)	0.016	0.08	0.4	2	10
Maximum shear stress (Pa)	0.003	0.016	0.083	0.414	2.068

$$\frac{d^2\theta}{dt^2} = \frac{\theta_{n-2} + \theta_n - 2\theta_{n-1}}{\Delta t^2}$$

$$\frac{d\theta}{dt} = \frac{\theta_{n-2} + 3\theta_n - 4\theta_{n-1}}{2\Delta t}$$

$$\theta = \theta_n$$

In terms of shear deformation, γ , we write the equation of motion as follows:

$$\gamma_{n\text{-}2}\left(\frac{\alpha}{\Delta t^2} + \frac{D}{2\Delta t}\right) - \gamma_{n\text{-}1}\left(\frac{2\alpha}{\Delta t^2} + \frac{2D}{\Delta t}\right) - \gamma_n\left(\frac{\alpha}{\Delta t^2} + \frac{3D}{2\Delta t} + E\right) = 0$$

where $\gamma_{n\text{-}1} = F_{\dot\gamma}\ \theta_{n\text{-}1}$, $\gamma_{n\text{-}2} = F_{\dot\gamma}\ \theta_{n\text{-}2}$, and $\gamma_n = F_{\dot\gamma}\ \theta_n$.The numerical resolution of this equation requires to leave free the three electromechanical parameters α, D, E, and two "initial" conditions $\gamma_{n\text{-}1}$ and $\gamma_{n\text{-}2}$. The values of the electromechanical parameters are reported on Table 1 and discussed in section 4. The two initial values are found roughly equal, for each step, which can be explained by the fact that they correspond to the imposed steady state at $\dot\gamma$ = 10^{-2} s^{-1} reached before each measurement.

APPENDIX B: TECHNICAL DATA

The geometry used in this study is a system of coaxial cylinders has the dimensions R_1 = 6 mm, R_2 = 6.5 mm, h = 18 mm, h_{eff} = 19.8 mm (effective height: taking end effects into account), F_σ 0.207*10^6 m^{-3}, $F_{\dot\gamma}$ = 12.52, and I = 0.57*10^{-6} kgm²

where F_σ = shear stress / torque = $\dfrac{R_2^2 + R_1^2}{4\pi h_{eff} R_2^2 R_1^2}$ and $F_{\dot\gamma}$ = shear rate / angular velocity = $\dfrac{R_2^2 + R_1^2}{R_2^2 - R_1^2}$. The measurement of torque is done through an angular compensation, with a sensitivity control which gives the maximum torque measured for each sensitivity. The maxima are given in Table 2.