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RHEOLOGY OF VISCOELASTIC FLUIDS: AN EXPERIMENTAL APPROACH

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Abstract: The present paper presents the rheology of viscoelastic fluids, using a Bohlin Gemini rheometer, with a con and plate geometry. The viscoelastic fluid proposed for study was a solution of 0.1% polyacrylamide (Separan AP 30) in sugar cane. The experimental data are obtained under simple shear and small-amplitude oscillatory shear flow conditions. The viscosity data, obtained from steady shear experiments, and the dynamic viscosity and storage modulus obtained from oscillatory shear experiments have been used to characterize the rheological behavior of the fluid.

INTRODUCTION

Rheology is “the study of the flow and deformation of all forms of matter” as defined by E.C. Bingham and M.Reiner in 1929 [1]. The main interest in rheology science is to analyse and describe the behaviour of relevant materials with intermediate properties between those of ideal solids and liquids, which cannot be described by classical theories.

It is a relatively young and multidisciplinary science that encompasses many different industrial areas of activity. It is quite straightforward to list situations where the deformation or the flow of matter (which depends on the rheological characteristics of the involved materials) determines the performance of a product, the effectiveness of a service and the rate of a manufacturing process. Thus, rheology is a very attractive, dynamic, highly multidisciplinary and fast-growing area of activity.

The understanding of polymeric fluids flows is of essential importance for several industry sectors, including plastic and food processing industries. The rheological response of viscoelastic fluids is quite complex, including combination of viscous and elastic effects and highly non-linear viscous and elastic phenomena.

Their constitutive entities are in interaction; the competition between the different energies generates structures at many different length scales in the fluid. This results in a diversity of macroscopic rheological behaviour, which can vary as a function of time, or as a function of the applied stress: elastic-plastic transition, shear thinning, shear thickening, thixotropy, [2], [3], [4].

The rheology of these soft materials has been the subject of many investigations. Experimentally, several methods are frequently used. Classical rheometry consists of globally shearing (stressing) the sample between two plates and recording the stress 0(strain or rotation velocity) applied by the sample onto the shearing plate. This allows the geometry and the frequency of the shear to be varied and is a very powerful technique to investigate materials with a linear response, [5].

However, in the case of nonlinear materials, the deformation field inside the sample does not decrease linearly with increasing distance from the shearing plate, like shown in Fig. 1. Models based on an inhomogeneous strain distribution inside the stressed fluid accurately account for the nonlinearities of the macroscopic stress-strain curve [6], [7].

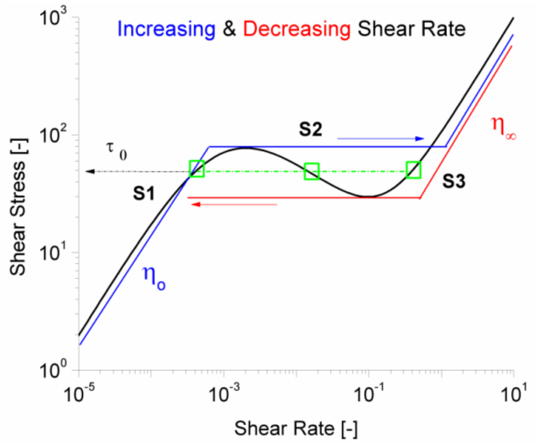


Figure 1. Non-monotonic flow curve. The yield stress corresponds to the "jump" in strain rate between the two stable domains (S1 and S2) on fluid's flow curve (with corresponding viscosity η_0 and η_∞).

The present investigation consists in a series of rheological measurements carried out with a controlled stress rheometer (Bohlin Gemini) using a cone-plate geometry.

Temperature control was achieved through a Peltier system, which guaranteed a thermal stability within ±0.1 ^{0}C.

Steady shear flow tests were done to measure the steady-state viscosity of the fluid. Linear oscillatory tests (upon verification of the linearity limit) and creeping tests were also performed to determine the viscoelastic response. The analysed sample is a solution with mass concentration 0.1% polyacrylamide (Separan AP 30) in sugar cane syrup. Finally, the rheological characteristics of the fluid have been obtained.

THEORY

For a given material (fluid, solid, soft solid) the rheological behaviour can be expressed mathematically trough a relation (constitutive equation) between two of its specific physical quantities. A constitutive equation emphasis the dependence of the applied stresses (τ), or forces to strains, or deformations (γ). Therefore, any decent analysis requires the knowledge and understanding of some fundamental concepts like viscosity, elasticity, viscoelasticity, stress, strain. Generally, practical processes consist of complex fluid flows difficult to analyse and describe analytically. Accordingly, the simple motions (like simple shear or extensional flow) are being used to determine rheological properties of fluids and soft solids, a commonly method being the approximation of experimental data obtained with different constitutive functions describing material behaviour.

When modelling a flow through a constitutive equation one important aspect is how the model parameters should be determined so that they may be suitable to be used in the process.

So far no constitutive model able to describe the fluid behaviour in every possible flow fields has been proposed. Therefore, any constitutive model has an area of performance within which the specified flow field may be explained satisfactory. Standard constitutive equations in literature are usually applied to some simple kinds of flows. From the rheological point of view, the viscoelastic fluids can be well described with the Cross model:

$$\eta = \eta_\infty + \frac{\eta_0 - \eta_\infty}{1 + (\lambda\dot{\gamma})^\alpha} \quad (1),$$

where η is the viscosity at any given shear rate $\dot{\gamma}$, η_0 and η_∞ are the asymptotic values of viscosity at zero-shear rate and infinite shear rate, λ is a constant time parameter with the dimensions of time and α is a dimensionless rate parameter indicating the degree of dependence of the viscosity on shear rate in the shear-thinning region.

Viscoelastic materials can be modelled in order to determine their stress or strain interactions as well as their temporal dependencies. These models, which include the Maxwell model, the Kelvin-Voigt model and the Standard Linear Solid model, are used to predict a material response under different loading conditions. Viscoelastic behaviour has elastic and viscous components modelled as linear combinations of springs and dashpots, respectively.

Viscoelasticity is studied using dynamic mechanical analysis, applying a small oscillatory strain and measuring the resulting stress. Purely elastic materials have stress and strain in phase, so that the response of one caused by the other is immediate. In purely viscous materials, strain lags stress by a 90 degree phase lag. Viscoelastic materials exhibit behavior somewhere in the middle of these two types of material, exhibiting some lag in strain, [2], [3], [7].

Complex Dynamic modulus G can be used to represent the relations between the oscillating stress and strain:

$$G = G' + iG'' \tag{2),}$$

where $i^2 = -1$; G' is the storage modulus and G'' is the loss modulus:

$$G' = \frac{\sigma_0}{\varepsilon_0}\cos\delta \tag{3}$$

$$G'' = \frac{\sigma_0}{\varepsilon_0}\sin\delta \tag{4),}$$

where σ_0 and ε_0 are the amplitudes of stress and strain and δ is the phase shift between them.

For viscoelastic fluids, the storage modulus and the loss modulus usually vary with the oscillations frequency.

Another specific property to the viscoelastic fluids is the viscoelastic creep: when such a fluid is subjected to a constant stress step, viscoelastic fluid experience a time-dependent increase with strain. Viscoelastic creep data can be presented by plotting the creep modulus (constant applied stress divided by total strain at a particular time) as a function of time. Below its critical stress, the viscoelastic creep modulus is independent of applied stress.

EXPERIMENTAL SETUP

The experimental set up used for this work was a Bohlin Gemini II rheometer, which represents an advanced range of compact research level instrument with “fluids to solids” capability (Figure 2), [8].

Figure 2. General view of the Bohlin Gemini II rheometer, [8]

The rheometer is optimized for both strain controlled and stress controlled operation, including a wide range of measurement geometries and accessories: temperature controller with Peltier device, forced gas oven with optional liquid nitrogen cooling, electrical heating and a range of fluids circulators. The latest Windows™ based operating software allows the Bohlin Gemini rheometer to be quickly and easily programmed for even the most complex test protocols. A user-friendly interface provides highly flexible test customisation and analysis to individual requirements. The rheometer is provided with a range of dedicated analysis software as standard including Time temperature superposition, Advanced data processing and Multiwave.

The main technical characteristic data are:
Torque range: 0.05 μNm to 200 mNm;
Position resolution: 50 nrad;
Frequency range: 1 μHz to 150 Hz;
Controlled speed range: 0.01 mrad s^{-1} to 600 rad s^{-1};
Normal force measurement range: 0.001 N to 20 N;
Step change in strain: <10 ms;
Temperature range with fluids circulator: - 40 ºC to 250 ºC.

RESULTS

The viscoelastic fluid proposed for study, Separan AP 30, which is a solution of 0.1% polyacrylamide in sugar cane, was initially characterized using a standard controlled stress measurement. The rheogram is plotted in Figure 3, showing a non-Newtonian behaviour.

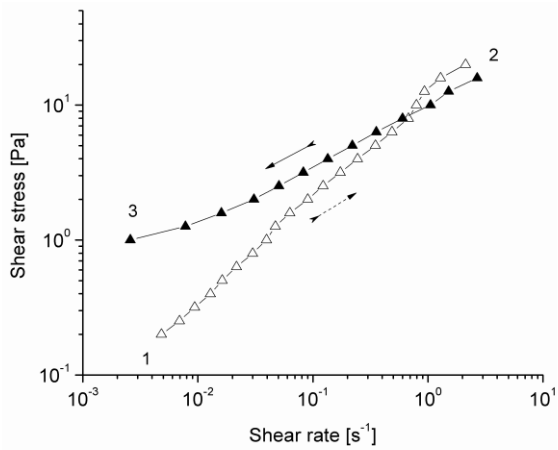


Figure 3. Rheogram of Separan AP30.

The experimental data where analyzed by the regression analysis method, [6], taking into account the Cross rheological model (Eq. 1). The values of the rheological parameters are:

the asymptotic value of viscosity at zero-shear rate: η_0 = 32.36 Pa.s;
the asymptotic value of viscosity at infinite shear rate: η_∞ = 7.25 Pa.s;
time parameter: λ = 6.773 s;
dimensionless rate parameter: α = 0.96;
statistical correlation coefficient: R^2 = 0.932.

Figure 4 presents the comparison between the experimental data and the viscosity fitting curve variation with shear rate.

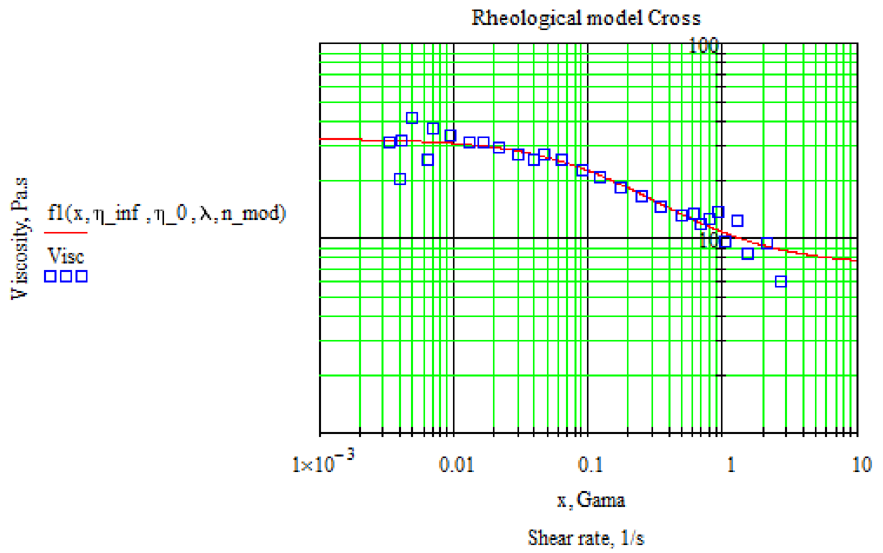


Figure 4. Comparison between experimental data and the theoretical curve of viscosity.

From oscillatory shear measurements the linear viscoelastic domain is first determined by a stress sweep (0.5 – 25 Pa) at a constant frequency (0.5 Hz). The storage *G*' and loss *G*'' modules are then measured by an angular frequency sweep in the range between 0.1 and 10 Hz, at a constant strain ε = 0.003. The results are presented in Figure 5.

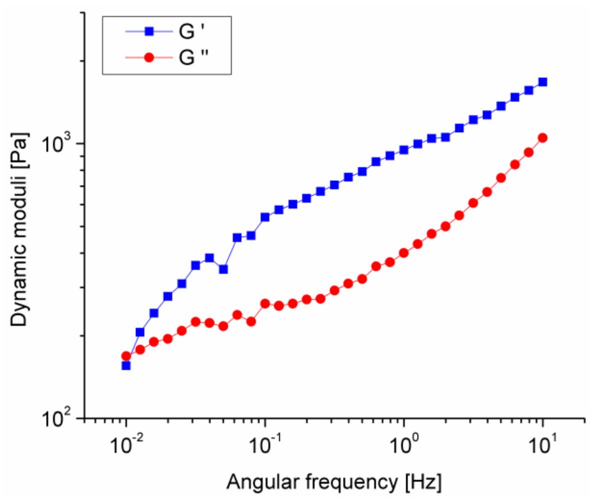
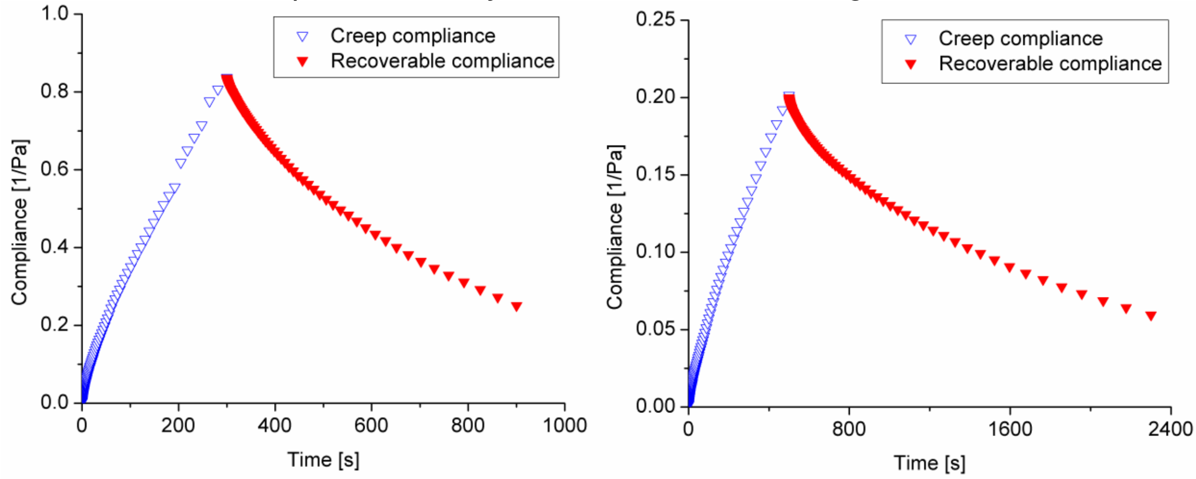


Figure 5. Variation of the storage and loss moduli versus frequency, at constant strain ε = 0.003.

The results for the creeping tests are presented in Figure 6, a and b, for two values of the imposed stress rate: 0.5 Pa and 0.8 Pa. It can be observed a clearly diminishing of the compliance, both for creep and recovery, once with the increasing of the stress rate.



a) ***b)***

Figure 6. Recoverable and creep compliance distributions for an applied stress of 0.5 (a) and 0.8 (b) Pa.

CONCLUSIONS

In this paper experimental methods were used to locally investigate the rheology of complex viscoelastic fluids. The viscoelastic modules of the fluid are deduced from the analysis of the motion of the probe in the fluid, taking into account a linear Maxwell rheological behaviour.

The following results have been obtained:

The viscosity of the suspension decreases when the applied shear increases. This decrease is compatible with a Cross model: $\eta = 7.25 + \frac{25.11}{1+\left(6.773\dot{\gamma}\right)^{0.96}}$;

The storage and loss modules increase with the increasing of the frequency at a constant imposed strain;

The creeping and recovery tests show a normal behaviour for a viscoelstic fluid; the compliance decrease with the increasing of the stress rate.

The experimental methods presented in this paper are promising for future investigation of complex fluids. However, when performing rheological measurements, one needs to considerate other rheological properties (surface tension, contact angle), sample homogeneity and external factors due to measuring setup (inertia, compliance, temperature gradients, etc).

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