EXPERIMENTAL INVESTIGATION OF THE YIELDING OF AN ELASTOVISCOPLASTIC MATERIAL

Rubens Rosario Fernandes, rubensr@utfpr.edu.br
Diogo Elias da Vinha Andrade, diogoandrade@utfpr.edu.br
Admilson Teixeira Franco, admilson@utfpr.edu.br
Cezar Otaviano Ribeiro Negrão, negrao@utfpr.edu.br

Research Center for Rheology and Non-Newtonian Fluids –CERNN, Postgraduate Program in Mechanical and Materials Engineering –PPGEM, Federal University of Technology-Paraná–UTFPR, Av. Sete de Setembro 3165, Curitiba 80230-901, PR, Brazil

Abstract. *Elastoviscoplastic materials present a transition from a gel-like to a liquid-like state induced by shearing: while the first is predominantly elastic, the second is predominantly viscous. The point that characterizes this transition is usually known as the yield point, which is traditionally associated to critical quantities such as a yield stress and a yield strain. The viscoelastic nature of elastoviscoplastic materials also leads to another characteristic transition from the linear to nonlinear viscoelasticity. In the present study, a commercial hair gel, which is a non-thixotropic elastoviscoplastic material, was tested in two rotational rheometers. Stress oscillatory amplitude sweeps at different frequencies were performed, aiming to characterize the linear viscoelastic strain and stress limits. This critical point was determined through a Fourier-Transform in the LAOS transient data for the oscillatory sweeps. Finally, the linear viscoelastic limiting point was compared to a critical point for yielding, which has been reported in literature to be associated with the crossover of G' and G'' in oscillatory amplitude sweeps. The results show that although the crossover strain and stress varied through the different experimental conditions, the linear viscoelastic limit strain and stress were remarkably constant. Also, the linear viscoelastic limit strain and stress coincided with the point in which irreversibilities were first observed in recovery experiments performed after successive steps on strain. This suggests that the yielding process of elastoviscoplastic materials might be associated to the onset of the nonlinear viscoelastic range rather than to the point in which the viscous behaviour overcomes the elastic one.*

Keywords*: rheology, elastoviscoplasticity, yield point, linear viscoelastic limit, critical quantities*

1. INTRODUCTION

Yield stress materials, such as drilling fluids, waxy crude oils and cosmetic gels and creams, have a wide range of industrial applications. These materials present a transition from a highly structured state at low strains, in which they deform reversibly, to an unstructured state when submitted to higher strains, when viscous irreversibilities take place (Chhabra e Richardson, 2011). An alternative point of view has been presented by some authors, that claim that most yield stress materials are, in fact, elastoviscoplastic materials (Ewoldt et al., 2010; Souza Mendes e Thompson, 2013). According to this point of view, yield stress materials present some level of viscous dissipation even below the yield stress, i.e., behave as viscoelastic materials both below and above the yield stress.

The gel-to-liquid transition induced by shearing is associated to a minimum shear stress that needs to be surpassed, known as yield stress. Several methods have been developed to evaluate the yield stress in rotational rheometers, such as the extrapolation of the flow curve in the limit of vanishing shear rates (Dimitriou et al., 2013), stress overshoots in constant shear-rate plateaus (Barnes e Nguyen, 2001), the crossover of G' and G'' in oscillatory amplitude sweeps (Rouyer et al., 2005), viscosity bifurcation in creep experiments (Coussot et al., 2002) and creep-recovery experiments (Nguyen e Boger, 1992). However, the yield stress assessed by these different methods usually leads to different values (Bonn et al., 2015), which brings an inherent difficulty in defining a critical point for the yielding of elastoviscoplastic materials. Recently, Tarcha et al. (2015) and Hou (2012) have brought to light the role of a critical strain for the yielding of waxy crude oils, which was reported to vary much less with the type of test performed than the yield stress. This critical strain can be associated to the yield strain of the material, since it is the strain observed in the point in which the yield stress is evaluated.

Elastoviscoplastic materials behave as viscoelastic solids in the limit of low strains (Frey et al., 2015). Therefore, elastoviscoplastic materials deform in the linear viscoelastic range prior to yielding. However, the linear-to-nonlinear viscoelastic transition in viscoelastic and elastoviscoplastic materials is much less reported in literature than the yielding of elastoviscoplastic materials. Kumar et al. (2012) observed that alumina suspensions left the linear viscoelastic region prior to yielding in oscillatory experiments. Additionally, some authors have reported some findings obtained with viscoelastic materials. Bird et al. (1987) observed that the linear-to-nonlinear viscoelastic transition occurs at a constant strain for a viscoelastic fluid in constant shear-rate start-up experiments. Golub and Fernatti (2005) and Riande et al. (2000), on the other hand, argue that the linear-to-nonlinear viscoelastic transition does not happen neither at a constant critical strain nor at a constant stress for viscoelastic materials under creep experiments. According to Riande et al. (2000), the linear-to-nonlinear viscoelastic transition depends on the time-scale of the experiment performed.

Considering the lack of works concerning the linear viscoelastic limit of viscoelastic and elastoviscoplastic materials and the association of this transition to the yielding of elastoviscoplastic materials, this work aims to investigate the relation between the linear-to-nonlinear viscoelastic transition and the yielding of an elastoviscoplastic fluid.

2. EXPERIMENTAL SECTION

The experimental investigation was conducted with a commercial hair gel (*Bozzano Extra Forte, Bozzano*®), which is an elastoviscoplastic Carbopol dispersion (Souza Mendes et al., 2014). The experiments were performed in two rotational rheometers: MARS III (Haake Co., Germany) and DHR-3 (TA Instruments, USA). The temperature of all the tests was controlled at 25 °C by Peltier systems assisted by thermostatic baths. A 35 mm diameter (MARS III) and a 40 mm diameter (DHR-3) parallel-plate geometries with a 1 mm gap and serrated surfaces to avoid wall slip (Dimitriou et al., 2011) were employed.

Two types of experiments were performed to estimate the yield point of the material. Firstly, 14 oscillatory stress amplitude sweeps at frequencies ranging from 0.05 to 5.0 Hz were performed, aiming to evaluate the yield point though the crossover of the storage and loss moduli, G' and G'' as suggested by Larson (1999). Three repetitions of the amplitude sweeps were performed for each one of the frequencies evaluate, summing up to 42 oscillatory experiments conducted. Then, creep and stress relaxation tests (constant shear stress and constant shear strain plateaus lasting 10 s each, respectively) followed by strain recovery steps at zero shear stresses were conducted so as to evaluate the yield point through the limit of reversibility of strains, as suggested by Nguyen and Boger (1992).

As described by Hyun et al. (2011), viscoelastic and elastoviscoplastic materials present a sinusoidal strain response when submitted to low amplitude sinusoidal stresses, i.e., in the linear viscoelastic range. When higher stress amplitudes are imposed, however, the material's response ceases to be in the linear viscoelastic range and the material enters in the LAOS (Large Amplitude Oscillatory Shear) regime. Thus, it is possible to define the limit of the linear viscoelastic range as the stress and strain amplitudes in which the strain response to a sinusoidal stress input ceases to be sinusoidal.

A Fourier transform can be performed in the strain response to each stress cycle to evaluate the different harmonics that compose the strain response signal. More details on the Fourier Transform method applied to oscillatory tests in rheology are described by Wilhelm et al. (1998). Therefore, each one of the n harmonics are described by a frequency (ω_n), by an intensity or amplitude (I_n) and by a phase angle (ϕ_n), such that the original strain response can be reconstructed as a function of time by Eq. (2.1).

$$\gamma(t) = \sum_n I_n \cos(n\omega_1 t + \phi_n) \tag{2.1}$$

The strain response should be well described by the first harmonic only if the material is below the nonlinear viscoelastic limit. Therefore, Hyun et al. (2011) propose that the nonlinearity in the signal response can be quantified by the intensity of the third harmonic relative to the intensity of the fundamental harmonic ($I_{3/1}$), as described in Eq. (2.2).

$$I_{3/1} = \frac{I_3}{I_1} \tag{2.2}$$

Thus, the material responds in the linear viscoelastic range as long as $I_{3/1}$ is smaller than a critical threshold, that will be adopted as $I_{3/1}$<0.5% as proposed by Hyun et al. (2011). This technique was proposed theoretically by Hyun et al. (2011) and adopted by Kumar et al. (2012) to determine the linear viscoelastic range of an elastoviscoplastic material, an alumina suspension, in oscillatory experiments.

3. RESULTS AND DISCUSSION

3.1 Oscillatory amplitude sweeps

The crossover of G' and G'' in oscillatory amplitude sweeps is traditionally associated to the yield point of yield stress materials (Andrade et al., 2015; Bonn et al., 2015; Carrier e Petekidis, 2009; Larson, 1999; Mason et al., 1996). However, associating this crossover to the yield point is inherently inaccurate, since the definitions of G' and G'' are valid only at the linear viscoelastic region and the material is invariably in the nonlinear range at the crossover (Bonn et al., 2015; Hyun et al., 2011). Thus, the use of the crossover as an estimate of the yield point must be done with caution, since it might not reflect the true nature of yielding although it is a point remarkably easy to identify. Because of that, the critical point defined by the crossover of G' and G'' will be defined as the *crossover point* instead of the *yield point* in the present work to avoid further misinterpretations.

Figure 1 presents the dynamic moduli, G' and G'', as a function of the stress amplitudes – Figure 1 (a) – and of the strain amplitudes – Figure 1 (b) – for the oscillatory amplitude sweeps conducted at one of the evaluated frequencies, 1.0 Hz. Keeping in mind the traditional method to determine the yielding, one can easily identify the two critical points, the crossover stress τ_c and the crossover strain γ_c in Figure 1 by the crossover of the two dynamic moduli. For this specific case, τ_c=276 Pa and γ_c=1,61 as shown by the red dashed lines. Also, the material ceases to respond in the linear viscoelastic range (characterized by the constant values of G' and G'') before the crossover of the dynamic moduli. This analysis was extended to the other frequencies evaluated, and the results are yet to be presented.

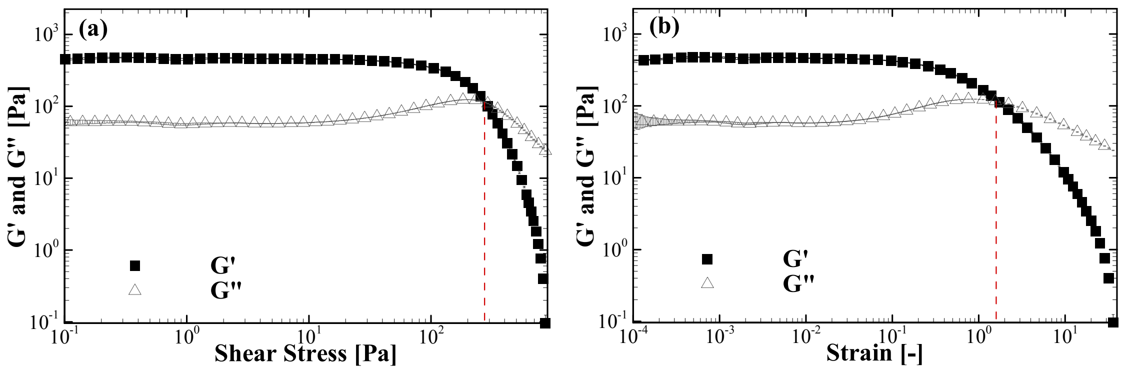

Figure 1 - Dynamic moduli as a function of the stress (a) and strain (b) amplitudes for the oscillatory amplitude sweep conducted at 1 Hz

The evaluation of the linear viscoelastic limit was performed by the Fourier Transform method. Figure 2 (a) presents the stress and the strain in the ordinates axis as a function of time in the horizontal axis recorded in one cycle of the sweep performed at 1.0 Hz that is in the nonlinear range. One can notice that although the shear stress imposed is sinusoidal, the resultant shear strain is non-sinusoidal and thus can be decomposed in a sum of harmonic sinusoids, according to the Fourier Series theory (Kreyszig, 2006). The amplitudes and frequencies of the sinusoids that compose the strain response can be obtained by a Fourier Transform, and are shown in Figure 2 (b). Figure 2 (b) presents the intensities of each one of the harmonics (I_n) relative to the intensity of the fundamental harmonic, (I_1), in the vertical axis, and the correspondent frequencies (ω_n) relative to the fundamental frequency (ω_1). The dashed line represents the limiting value of the relative intensity of the third harmonic, $I_{3/1}$, for the linear viscoelastic range, as proposed by Hyun et al. (2011). As can be seen, $I_{3/1}$>0.5% and thus the material is in the nonlinear viscoelastic range for this specific cycle.

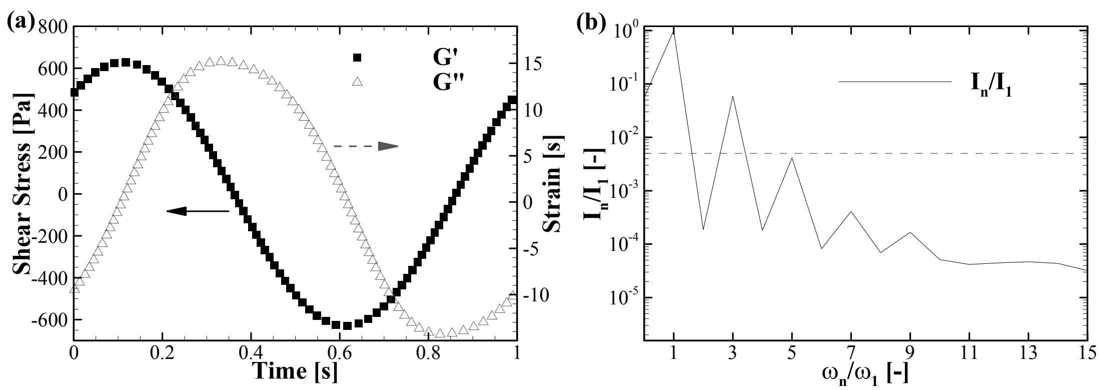

Figure 2 – Shear stress and shear strain as a function of time for one cycle in the non-linear range of the oscillatory amplitude sweep with 1.0 Hz frequency (a); and intensities of the different harmonics that compose the strain output signal relative to the fundamental harmonic intensity as a function of the correspondent frequencies relative to the fundamental harmonic frequency (b)

This technique was employed to the cycles performed at all the frequencies evaluated so as to assess the linear viscoelastic limit strains and stresses. Thus, Figure 3 (a) presents the linear viscoelastic limit strains, γ_c^{lv}, evaluated through the Fourier Transform, and the crossover strain γ_c evaluated at the crossover as a function of the imposed frequencies. Analogously, Figure 3 (b) presents the critical stresses τ_c^{lv} and τ_c in the vertical axis as a function of the imposed frequencies. The points represent the average of the critical stresses and strains for the three repetitions performed at each one of the frequencies evaluated, whilst the error bars represent the standard deviation of the measured quantities. The results presented in Figure 3 indicate that the transition from the linear-to-nonlinear viscoelasticity occurs at strains and stresses lower than those associated to the crossover in all the frequencies evaluated. Also, the crossover stress, τ_0, depends on the applied frequencies, and thus on the time-scale of the experiments.

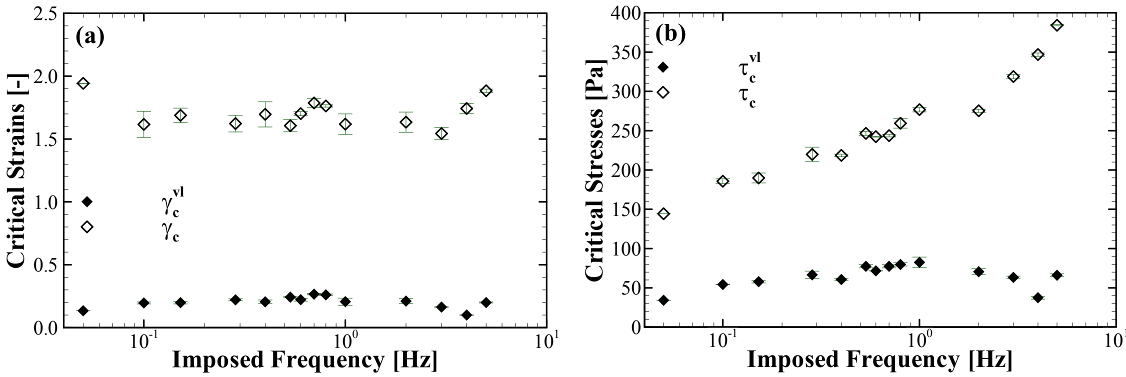

Figure 3 - Critical strains (a) and critical stresses (b) as a function of the imposed frequencies for the oscillatory experiments

3.2 Recovery Experiments

In both experiments performed above, the material left the viscoelastic range before reaching the "yield point" defined by the crossover of G' and G''. An alternative approach to evaluate the yield point is to perform recovery experiments. According to Nguyen and Boger (1992), this type of experiment assesses the yield point by evaluating the limit of reversibility of the material's structure when submitted to increasing loads. This is a rigorous way of measuring the yield point, since it takes into account the point above which the first irreversibilities start to take place on the material's microstructure. Therefore, this method can also be understood as a method to evaluate the yield point through the evaluation of the reversibility limit. Constant strain plateaus, γ_p, ranging from 10^{-3} to 10^{-1} were imposed for 10 seconds. Steps of zero stress lasting 30 min, during which the strain recovery was measured, followed each strain plateau. Therefore, the material is strained during the strain plateaus, and is allowed to rest during the zero stress steps. Since the intensity of the strain plateaus increases after each strain/recovery cycle, the loading condition above which strain irreversibilities start to take place should be evidenced by the material's inability to completely return to its original position. The resultant shear strain as a function of time is presented in Figure 4. When strains lower than 0.04 are imposed, the material's strain recovery is complete and nearly instantaneous. However, when strains higher than 0.04 are imposed, the recovery ceases to be complete in the time scale evaluated.

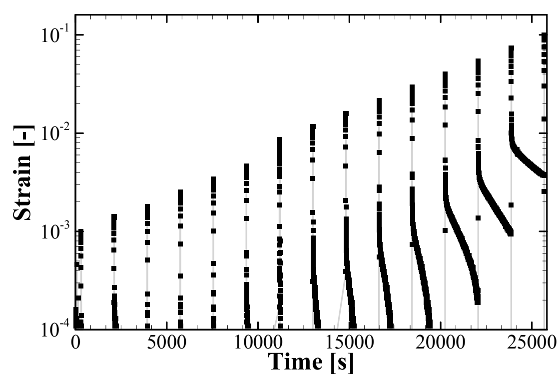

Figure 4 – Strain as a function of time for the recovery experiments performed with imposed strain (a) and stress (b) plateaus

One can evaluate the fraction of the strain recovered in each one of the recovery experiments performed through Eq. (2.3), in which γ_{rec} is the percent fraction of strain recovered, γ_f is the final strain of each strain plateau, and γ_{res} is the residual strain that is observed at the end of each recovery step.

$$\gamma_{rec}[\%] = \frac{\gamma_f - \gamma_{res}}{\gamma_f} \cdot 100 \tag{2.3}$$

Thus,

Figure 5 presents the fraction of strain that is recovered, Eq.(2.3), as a function of the final strains,
Figure 5 (a), and stresses,
Figure 5 (b), observed at the end of each strain plateau. The horizontal dashed line represents the limit of 99% of recoverability, whereas the vertical dashed lines indicate the yield stresses and strains evaluated with the recovery experiments with imposed strains. The experiments performed revealed that the point above which the reversibility of strains becomes lower than 99% corresponds to a critical strain of 0.054 and a critical stress of 18.4 Pa.

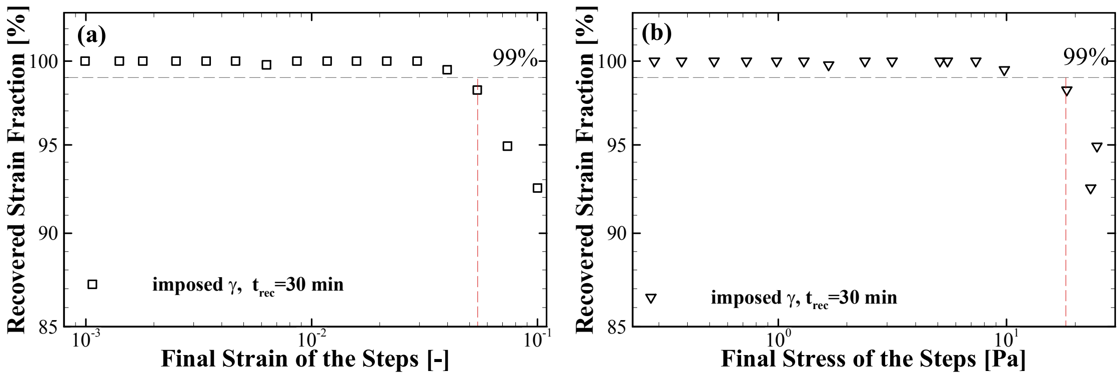

Figure 5 – Percentage of strain recovered as a function of the final strains (a) and stresses(b) of the loading steps plateaus of the recovery experiments

3.3 Association between the linear viscoelastic limit and the yield point

The evaluations of the crossover point and of the linear viscoelastic limit were obtained through oscillatory amplitude sweeps, as described in Section 3.1. Furthermore, different time scales – represented by the several frequencies for the oscillatory sweeps – were evaluated.

Therefore, it is finally possible to compare the estimates of the crossover stresses and strains obtained through the oscillatory sweeps and the yield stress and strain obtained with the recovery experiments with the estimates of the linear viscoelastic limit stresses and strains obtained with the oscillatory sweep experiments. Figure 6 (a) presents the critical strains as a function of the frequencies imposed in the oscillatory experiments, whereas Figure 6 (b) presents the critical stresses as a function of the frequencies imposed. The error bars represent the standard deviation of the three repetition of each one of the experimental conditions. The continuous line stablishes the value of the yield stresses and strains obtained with the recovery experiments with imposed strains.

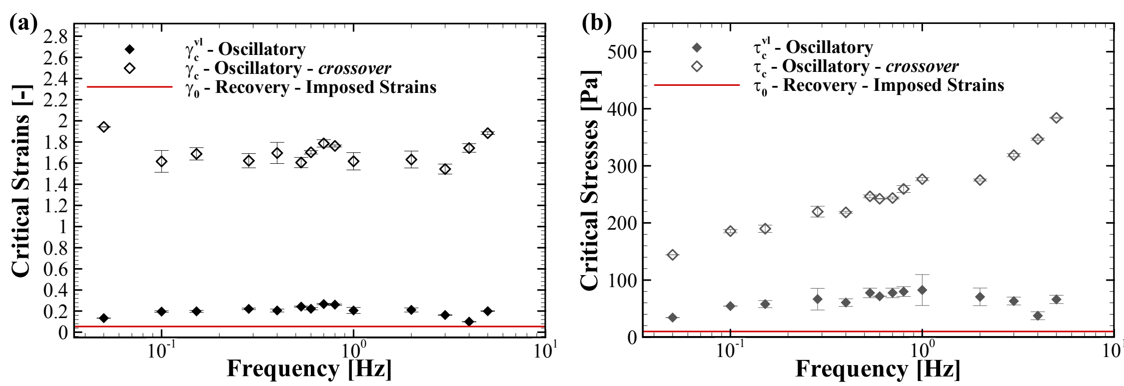

Figure 6 – Critical strains (a) and stresses (b) as a function of the normalized characteristic times for the different experiments conducted with the Carbopol dispersion

There are several features of Figure 6 that deserves attention. First of all, one can notice that the quantities γ_c and τ_c that define the yield point according to several works in literature – i.e., the crossover of G' and G'' - are considerably higher than the quantities that define the linear viscoelastic limit, γ_c^{vl} and τ_c^{vl}, for the oscillatory experiments. This indicates that the material leaves the linear viscoelastic region prior to the loading condition that is normally associated to yielding. Furthermore, the values of γ_c and τ_c varied considerably with the time scales of the oscillatory experiments (i.e., the imposed frequencies), whilst γ_c^{vl} and τ_c^{vl} were remarkably constant, regardless of the time scale of the experiments performed. Finally, the reversibility limit determined through the recovery experiments was in the same order of magnitude of the linear viscoelastic limit stresses and strains, γ_c^{vl} and τ_c^{vl}, evaluated through the oscillatory experiments. Although the values of γ_0 and τ_0 measured in the recovery experiments are still lower than the values of γ_c^{vl} and τ_c^{vl}, the linear viscoelastic limit is much more close to the yield point measured in the recovery experiments than the crossover point assessed by the crossover of G' and G'' in the oscillatory tests. Therefore, it is plausible to suppose that the linear-to-nonlinear viscoelastic transition is probably associated to the region in which the first viscous dissipations start to take place in the material's microstructure, which are characterized by the onset of irreversibilities on the strain recovery.

This indicates that the crossover of G' and G'' must be used with caution as estimates of the yield point, since they represent the material's behavior at a loading condition in which the material's straining is already irreversible. Therefore, the linear viscoelastic limit appears to be a feasible candidate to estimate the yield point with a more rigorous approach.

4. CONCLUDING REMARKS

The current work aimed to provide a new insight on the relation between the nonlinear viscoelasticity and the yielding of an elastoviscoplastic material. Although there are several methods established to evaluate the yield point of elastoviscoplastic materials, the determination of the linear viscoelastic limit is much less reported in literature. Therefore, oscillatory stress amplitude sweeps were performed to assess the crossover point (which is traditionally associated to the yield point) and the linear viscoelastic limit of a commercial hair gel. The crossover point was determined as the crossover of the dynamic moduli G' and G'' in the oscillatory tests. The linear viscoelastic limit, on the other hand, was determined through a Fourier transform on the oscillatory test's results. Additionally, recovery experiments were also performed, in which the yield point was estimated as the loading condition above which the strain recovery was lower than 99%. The main conclusions of the paper might be summarized as:

i. In the oscillatory experiments, the quantities that define the linear viscoelastic limit - i.e., τ_c^{vl} and γ_c^{vl} - were lower than the crossover stresses and strains, which are used as estimates of the yield point according to literature;
ii. The values of the crossover strains and stresses estimated through oscillatory tests presented a considerable dependence on the imposed frequencies of the experiment;
iii. The values of the linear viscoelastic limit strains and stresses, on the other hand, presented a smaller dispersion among the different imposed frequencies for the oscillatory experiments;
iv. The linear viscoelastic limit evaluated with the oscillatory experiments was of the same magnitude of the reversibility limit estimated through the recovery experiments, indicating that the linear viscoelastic limit correspond to the onset of irreversibility's on the material's microstructure.

Considering the above discussion, it is plausible to deduce that that irreversible deformations start to take place above the linear viscoelastic limit, which can be understood as an estimate of the yield point of the material.

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