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Connecting Large Amplitude Oscillatory Shear Rheology to Steady Simple Shear Rheology and Application to Biomass Slurries

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Abstract:
Large amplitude oscillatory shear (LAOS) rheology is often performed in order to complement steady simple shear (SSS) rheology, i.e., probe rheological properties of materials that cannot be not observed with SSS alone. However, it is difficult to measure the SSS rheology of some problematic materials due to fracture and ejection, and LAOS may alleviate these issues, at least partially. Therefore, it is of interest to obtain SSS rheology information from LAOS measurements. We show that a constitutive modeling approach may be used to unify the analysis of LAOS data obtained from different viscometric geometries and modes of control and that the LAOS data may be used to predict SSS profiles. A model elastoviscoplastic material, a Carbopol solution, was used to validate the approach experimentally. LAOS rheometry of problematic biomass slurries was also performed, and the SSS profiles for the slurries were predicted with more confidence than could be obtained from SSS measurements directly.

Key words:
LAOS, elastoviscoplastic, yield stress, Carbopol, biomass slurry

1 *INTRODUCTION*

Many rheometric methods have been developed to probe the rheological behavior of complex fluids and soft solids, including unidirectional shear-stress sweeps, creep and relaxation tests, and small-amplitude oscillatory shear (SAOS), to name a few [1]. Each of these methods were typically developed to probe just one or two defined rheological properties, e.g., yield stress, loss and storage moduli, and shear-rate-dependent viscosity prole. Recently, large-amplitude oscillatory shear (LAOS) is receiving considerable attention because it has the potential for probing several rheological properties of a material at once with a relatively small set of experiments [2, 3]. In addition, LAOS may alleviate some of the problems of fracture and ejection that occur in problematic materials when exposed to the unbounded deformations of steady simple shear (SSS) [4]. However, the analysis of the complex, nonlinear oscillatory profiles that result from LAOS is not straightforward, and a few differing approaches have been proposed for obtaining material functions from LAOS data without first presuming a constitutive model [2, 5–10]. This body of work has advanced the experimental methodology, qualitative interpretation, and quantitative analysis of LAOS considerably. Nonetheless, several issues remain, including the different behaviors that are observed between strain- and stress-controlled measurements and between uniform- and nonuniform-shear viscometric geometries (e.g., cone-and-plate vs. parallel-plate). These differences pose significant challenges to the leading quantitative analysis methods of analyzing LAOS rheology data, which so far have been limited to the uniform-shear case.

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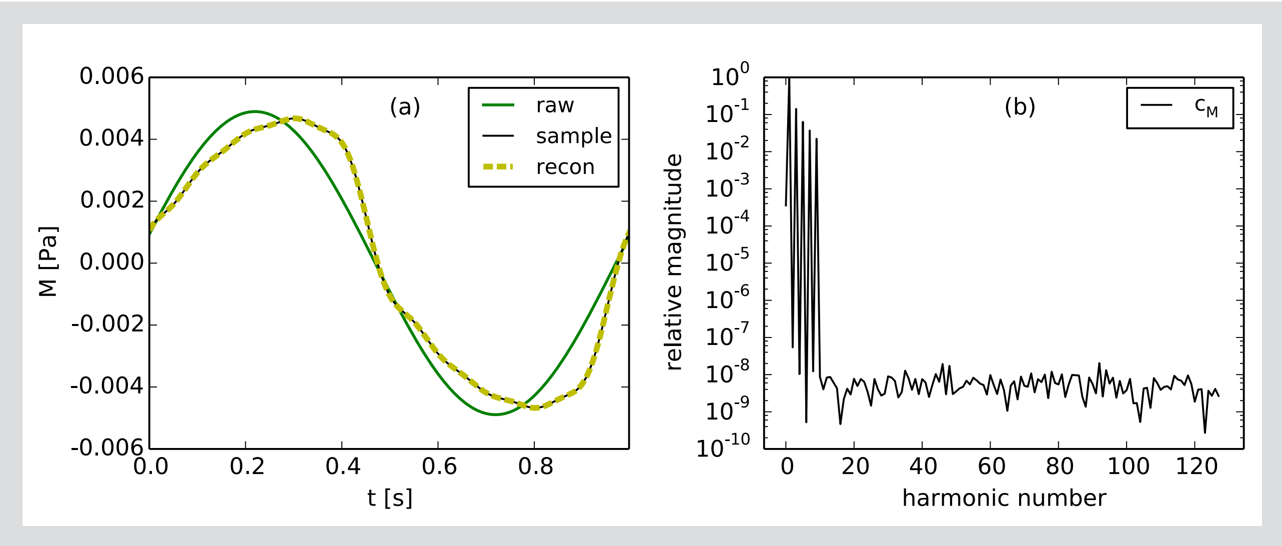

Figure 13: Inertial correction for torque-controlled parallel-plate measurement of Carbopol, Γ_1 = 67.9 and Bi^{-1} = 2.7. (a) raw, sample, and reconstructed torque signals (sample and reconstructed signals overlap); (b) normalized magnitude of the sample torque Fourier coefficients.

The Fourier coefficients for the conventional signal, $f(t)$, where $t = \hat{t} + t_o$, can then be determined by

$$c_n = \hat{c}_n e^{-in\omega t_o} \tag{19}$$

The effect of instrument inertia, I, may be accounted for by subtracting the torque due to the inertia from the raw torque signal:

$$M_S = M_R - I\frac{d^2\theta}{dt^2} \tag{20}$$

In order to evaluate Equation 20, it is necessary to obtain the second derivative of the displacement signal. We found it numerically favorable to compute the second derivative of the Fourier series representation of displace- ment, specifically,

$$\frac{d^2\theta}{dt^2} \approx -\sum_{n=-N}^{N} (n\omega)^2 c_{\theta n} e^{in\omega t} \tag{21}$$

Again,it is necessary to truncate the series to reduce noise. In fact, high-frequency signal variation, whether real or artifact, is magnified by differentiation [33]. This is exhibited in the Fourier representation (Equation 21) by the coefficients scaling with $(n\omega)^2$. Because we are interested in the inertial contribution from the primary oscillatory displacement signal, and not from the high-frequency oscillations, we truncate the series based on the values of $c_{\theta n}$. By subsequently evaluating Equation 21 at the same sampling time points as for M_R, it was straightforward to obtain the sample torque from Equation 20. We chose to obtain the sample torque from the raw torque before smoothing the torque.

Examples of (phase-shifted) noisy displacement and torque signals obtained from a displacement-controlled LAOS experiment are shown in Figures 12a and 12b. Normalized Fourier coefficients for the displacement and sample torque signals are shown in Figure 12c. Although we did not attempt to probe the source of the persistent broad-band noise, we suspect it is due to the limitations of the displacement feedback-control system.By dropping Fourier series terms beyond 10 resulted in the smoothed reconstructed signals shown in Figures 12a – b.

For a torque-controlled measurement, the instrument implements a sinusoidal raw torque and measures the displacement signal. However, after accounting for the instrument inertia, the sample torque may not be sinusoidal, as illustrated in Figure 13. Dimitriou et al. [5] provided analysis for determining an upper bound on stress amplitude so that inertia was negligible, maintaining a sinusoidal sample torque. In the current work, a truncated Fourier series was used to represent the imposed sample torque when performing model simulations of torque-controlled LAOS rheometry. Hence, perfectly sinusoidal imposed signals were not necessary for the constitutive modeling approach used here.

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