PHYSIC 607: Experiments in Squishy Physics Fall 2025 Lab 2: Bulk Rheology

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Abstract

Rheology is the study of how materials deform and flow under applied stress. It bridges the behavior of ideal solids, which store energy elastically, and ideal fluids that dissipate energy through viscous flow. Rheological measurements were performed on glycerol and agarose samples. These measurements were collected using a TA Instruments DHR2 rheometer and Trios software. Each sample underwent oscillatory shear, creep, and flow tests to characterize viscoelastic properties. The experimental values for the storage modulus $G'(\omega)$ and the loss modulus $G''(\omega)$ were compared to theoretical expectations for Newtonian and viscoelastic materials.

Bulk Rheology 1 BACKGROUND

1 Background

Visscoelastic response of materials can be described through measurements of storage and loss moduli G' and G'', respectively. Glycerol, a Newtonian fluid, was expected to exhibit a dominant viscous response where G'' >> G' while agarose, a gel-like material, was expected to exhibit a dominant elastic response with G' >> G''. Theoretical models predict that in the linear viscoelastic regime, these properties remain constant with strain amplitude, allowing for accurate characterization of material response under small deformations.

Bulk rheology is one of the most effective ways to study the viscoelastic properties of materials. Specifically, shear rheology is most effective with studying the differences in Hookean solids, Newtonian and non-Newtonian fluids.

Utilizing the stress of a material,

$$\sigma(t) = \gamma_0(G'(\omega)\sin(\omega t) + G''(\omega)\cos(\omega t))$$

where γ_0 is the initial stress, $G'(\omega)$ is the elastic (storage) module, $G''(\omega)$ is the viscous (loss) module and is the angular frequency, a host of information can be determined about the material through the use of oscillatory shear, creep and flow rheology studies.

In oscillatory shear rheology, by holding the angular frequency constant and conducting an amplitude sweep, the linear viscoelasticity portion of the material can be studied to identify the maximum oscillatory shear before the material deforms. By holding the amplitude constant and varying angular frequency, the material's relaxation times can be found.

Creep rheology techniques can be used to determine the relaxation time, τ , where the tradeoff of storage modulus and the loss modulus is.

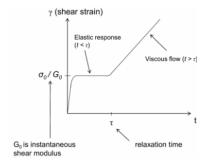


Figure 1: Shear Strain against time

Before the relaxation time, the storage module is greater than the loss module and thus material acts like a Hookean solid. After the relaxation time, the loss module out performs the storage module and the material acts like a fluid. For Hookean solids, the relaxation time is relatively big compared to that of fluids.

For viscous fluids, stress can be expressed as

$$\sigma(t) = \eta \frac{dy}{dt}$$

where is the viscosity of the fluid and $\frac{dy}{dt}$ is the change in strain stress with respect to time. For Hookean solids,

$$\sigma(t) = G\gamma$$

where G is the shear modulus.

For Newtonian fluids, when a stress is applied, the viscosity does not change which contributes to their property that their "phase" does not change with respect to how it is handled. For non-Newtonian fluids, viscosity does change with respect to the change in stress with respect to time. If the viscosity decreases as stress increases, the material will undergo shear thinning and will flow more freely as exemplified through toothpaste or lotion. On the other end of the spectrum, if viscosity increases, the material will go through shear thickening and tighten up commonly seen in oobleck.

2 Materials and Methods

Three separate materials were prepared and subjected to standard oscillatory shear rheology tests.

The first material tested was agarose in gel form. The agarose was heated to transition it into its liquid phase. Using a $500\,\mu\text{L}$ pipette, it was then placed on the rheometer's temperature controlled plate which was then set to a standard $23\,^{\circ}\text{C}$ which was used for all tests and materials. Careful thought was used to remove air bubbles from the sample. The liquid agarose was then given time to resolidify back into a gel. Once the agarose was in its final phase, the geometry was set to $311.0\,\mu\text{m}$ and rotated to evenly distribute the sample across the entire surface of the geometry.

The glycerol was loaded onto the rheometer using a $350\,\mu\text{L}$ pipette. Air bubbles were then removed from the sample using the tip of the pipette. The geometry was then lowered to $212\,\mu\text{m}$ and the temperature controlled plate was set to a standard temperature of $23\,^{\circ}\text{C}$ and the geometry was rotated to evenly distribute the sample across the entire surface of the geometry.

The final material, mayon naise, was spooned onto the rheometer. The geometry was lowered to a height of $1200\,\mu\mathrm{m}$ and the temperature controlled plate was set to a standard temperature of $23\,^{\circ}\mathrm{C}$ and the geometry was rotated to evenly distribute the sample across the entire surface of the geometry.

The following standard parameters were set for each test against the individual samples. Note that since Agarose acts as a Hookean solid, a flow test was not conducted.

Oscillatory Amplitude Sweep Test				
Material	Agarose	Glycerol	Mayonnaise	
Oscillation Dis-	1.6×10^{-5} - $9.9 \times$	4.3×10^{-6} - $4.2 \times$	6.0×10^{-4} - $6.3 \times$	
placement (rad)	10^{-4}	10^{-3}	10^{-3}	
Angular Fre-	10.00	10.00	10.00	
quency $\frac{rad}{s}$				
Oscillation	0.10 - 6.35	0.04 - 40.00	0.10 - 10.00	
Strain (%)				
Temperature °C	23.00	23.00	23.00	
Test Duration	54	102	68	
(s)				

 Table 1: Oscillatory Amplitude Sweep Test Parameters

Oscillatory Frequency Sweep Test				
Material	Agarose	Glycerol	Mayonnaise	
Oscillation Dis-	1.2×10^{-4}	1.1×10^{-4}	5.9×10^{-4}	
placement (rad)				
Angular Fre-	1.00 - 100.00	0.10 - 100.00	0.10 - 100.00	
quency Range				
$\frac{rad}{s}$				
Sweep Type	Linear	Linear	Linear	
Temperature °C	23.00	23.00	23.00	
Test Duration	67	181	178	
(s)				

Table 2: Oscillatory Frequency Sweep Test Parameters

Creep Test				
Material	Agarose	Glycerol	Mayonnaise	
Strain (%)	0 - 0.24	0 - 636.74	1.40 - 0.087	
Temperature °C	23.00	23.00	23.00	
Test Duration	59	58	29	
(s)				

Table 3: Creep Test Parameters

Flow Test				
Material	Agarose	Glycerol	Mayonnaise	
Shear Rate (s^{-1})	N/A	0.10 - 100.00	78.72	
Stress (Pa)	N/A	0.094 - 52.11	0.1 - 400.00	
Temperature °C	23.00	23.00	23.00	
Test Duration	N/A	526	666	
(s)				

Table 4: Flow Test Parameters

3 Experimental Results and Analysis

3.1 Agarose

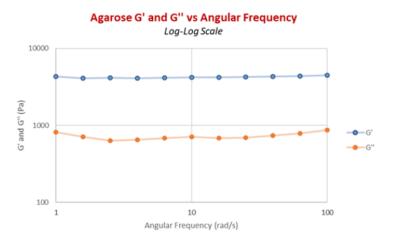


Figure 2: Storage modulus and viscous modulus of Agarose against angular frequency for an oscillatory frequency test.

Agarose behaves as solid with a larger elastic module than viscous G' >> G'' and no G' = G'' crossover. Deborah number De is the relaxation time/the observation time and $D_e = 1$ for all of , which confirms the elastic behavior observed. Since there is no plateau observed, there is no measurable η_0 .

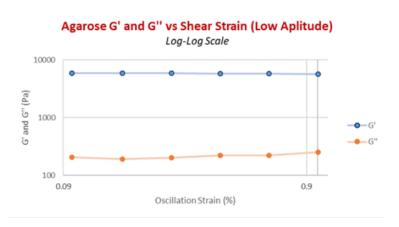


Figure 3: Storage modulus and viscous modulus of Agarose against oscillation strain for an oscillatory amplitude sweep test.

Linear viscoelastic range: 0.1-1% strain. The graph is flat at around $G' \approx 5.8-6.0$ kPa. Phase angle $\delta \approx 0.03-0.04$. Onset of nonlinearity is approximately 2–3% strain. Fracture of agarose begins around 12% strain and crossover G' = G'' occurs around 14% strain. Yield stress estimate is approximately 0.5-0.7 kPa.

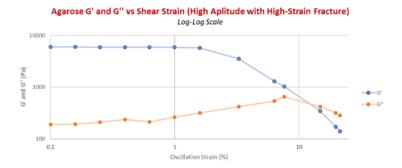


Figure 4: Storage modulus and viscous modulus of Agarose against shear strain for an oscillatory sweep frequency test with high strain fracture.

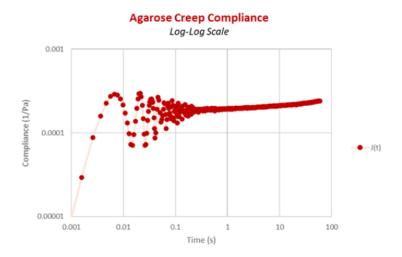


Figure 5: Compliance Agarose against time for a creep test.

The short-time regime (t < 0.02 s) is ignored due to it being dominated by instrumental artifacts and not conducive for determining compliance J(t). The behavior is that of a viscoelastic solid with no evident $J(t) \approx t/\eta$ which would be seen in a viscous material.

In conclusion, our results are consistent with what one would expect for an agarose gel under the conditions of the experiment; the sample exhibited a predominantly elastic response. No true plateau is observed in the frequency sweep so there is no measurable value for zero shear viscosity η_0 .

The complex shear modulus is expressed as $G^*(\omega) = G'(\omega) + iG''(\omega)$ where $G'(\omega)$ is the elastic (storage) modulus and $G''(\omega)$ is the viscous (loss) modulus. Across the frequency tested, we observed that G' >> G'', which confirms a strong elastic response opposed to a freely-flowing viscous liquid where G'' would be dominant. Overall, all data confirm that agarose behaves as an elastic gel with negligible viscous flow.

3.2 Glycerol

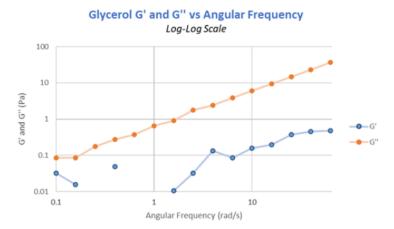


Figure 6: Storage modulus and viscous modulus of Glycerol against angular frequency for an oscillatory frequency test.

There is noise dominant behavior at low frequency. However, the plot clearly shows that glycerol's response is liquid-like, with viscous energy dissipation dominating elastic storage (G'' >> G') and a large phase angle of $\delta = \frac{G''}{G'} >> 1$. There is no crossover between G' and G'', indicating no measurable elastic regime. For a Newtonian fluid, G' = 0 and $G'' = \eta_0 \omega$. At low frequency $(0.1 \frac{rad}{s})$, our results give zero shear viscosity of

$$\eta_0 = \frac{G''}{\omega} = 0.859 \, Pa \cdot s$$

The Deborah number D_e is < 1 for all ω , which confirms the viscous behavior observed.

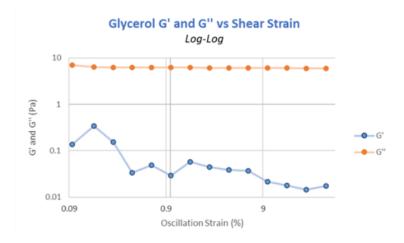


Figure 7: Storage modulus and viscous modulus of Glycerol against oscillation strain for an oscillatory amplitude sweep test.

G'' is linear with the most linearity happening between approximately 0.16% to 25.27% strain, while G' remains near zero and is noise dominated at lower strain. There is no

yield stress. Glycerol exhibits Newtonian fluid behavior throughout the experiment with no plateau or drop indicating yielding. Stress scales as $\sigma_0 = G''\gamma_0$ which is consistent with a purely viscous response.

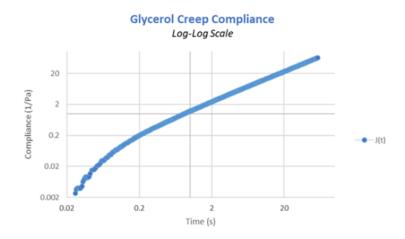


Figure 8: Compliance Glycerol against time for a creep test.

The creep plot shows we have viscous flow with negligible elasticity. Note: data for t < 0.05 s was excluded from analysis due to instrumental noise. The region 0.1 - 1 s represents the transition, and for $t_1 s$ the creep compliance J(t) grows linearly with time (slope of $\frac{dJ}{dt}$), showing no elastic plateau. Using a slope over a long time, where J(t) is consistent, 10-30 s was chosen

 $\frac{dJ}{dt} = 1.16 \left(Pa \cdot s \right)^{-1}$

SO

$$\eta_0 = \frac{1}{1.16 (Pa \cdot s)^{-1}} = 0.86 Pa \cdot s$$

This value agrees with the zero shear viscosity η_0 determined from the frequency sweep results. The frequency sweep showed G'' >> G', which indicates viscous-dominated behavior. This is consistent with the complex shear modulus $G^*(\omega) = G'(\omega) + iG''(\omega)$, and our creep results confirm that G'' dominates over G' which verifies glycerol's liquid-like response.

The flow test shows Newtonian behavior since eff remains nearly constant with shear rate, making stress proportional to shear rate. Glycerol, therefore, behaves as a Newtonian fluid, with eff independent of shear rate.

In conclusion, all rheological tests confirm that glycerol behaves as a Newtonian liquid at approximately 23°C. For the amplitude sweep, G'' remained linear between about 0.16% to approximately 25% strain, while G' was close to zero showing no yield stress. The frequency sweep showed G'' >> G' with no crossover, indicating a liquid-like response dominated by viscous energy dissipation. The Deborah number was < 1 across all frequencies. Creep measurements showed a linear increase in compliance J(t) with time and no elastic plateau. Both the oscillatory and creep measurements obtained zero shear viscosity $\eta_0 \approx 0.86 \, Pa \cdot s$. The flow test showed the effective viscosity remained constant ($\approx 0.6 \, Pa \cdot s$) over the shear rate

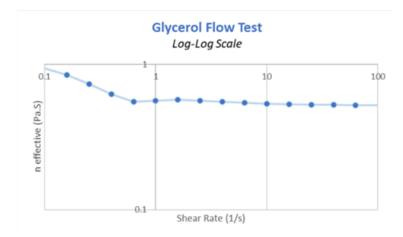


Figure 9: Viscosity of Glycerol against shear rate for a creep test.

which confirms that stress is proportional to shear rate. Together, these results demonstrate that glycerol exhibits purely Newtonian liquid behavior with negligible elasticity.

3.3 Mayonnaise

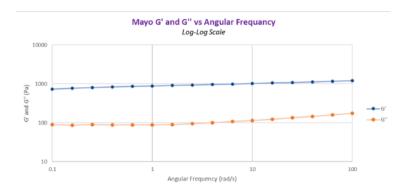


Figure 10: Storage modulus and viscous modulus of Mayonnaise against angular frequency for an oscillatory frequency test.

For our frequency sweep plot, it shows Mayo as a solid-like material with G' > G''. There is also no relaxation time crossover where G' = G''. G' does not show ω^2 scaling at low ω , so $G'' \approx \eta_0$ is not satisfied. η_0 cannot be determined from the data obtained. Deborah number is > 1 for this experiment so the results are consistent with a solid-like response.

The graph for Mayo vs. Shear strain shows a linear regime strain 0.2-1.6%. G' stays approximately 900-916 Pa and G'' around 87-93 Pa with <1-2% drift up to 1.6% strain. The graph shows no clear yield stress because of no apparent G'' peak. However, onset of non-linearity begins around 2.5-4% as G' drops and phase angle rises. The estimated yield onset stress can be calculated by

$$\sigma = G_0' \gamma \tag{1}$$

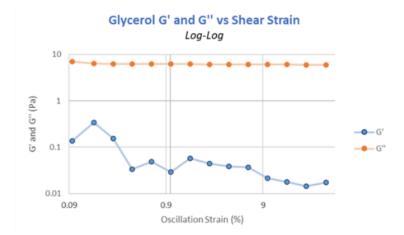


Figure 11: Storage modulus and viscous modulus of Mayonasel against oscillation strain for an oscillatory amplitude sweep test.

$$\approx 22.75 \, Pa$$

for the lower limit and

$$\approx 36.40 \, Pa$$

for the upper limit. Full yield is not reached by the experimental 10.4% strain.

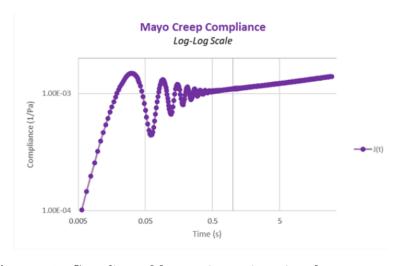


Figure 12: Compliance Mayonnaise against time for a creep test.

The creep graph shows that elastic response dominates the experiment for Mayo. J(t) rises quickly. Slope is 1, so there is no steady viscous flow within the time frame of experiment (30 s).

The flow graph for Mayo shows strong non-Newtonian shear-thinning behavior. Viscosity drops from around $787 \, Pa \cdot s$ at $0.1 \, s^{-1}$ to about $0.92 \, Pa \cdot s$ at $400 \, s^{-1}$. This result matches $G * (\omega)$; elastic-dominant response and no terminal flow in the experimental window.

Overall, our results show Mayo exhibiting a primarily solid-like viscoelastic response. The

Bulk Rheology 4 DISCUSSION

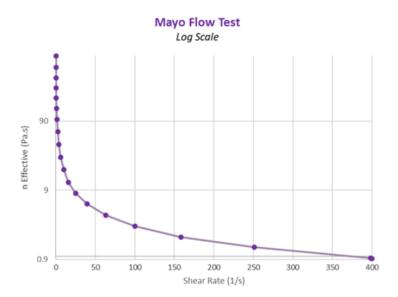


Figure 13: Viscosity of Mayonnaise against shear rate for a creep test.

amplitude sweep shows a linear regime up until about 1% strain, with a yield onset near 3%. The frequency sweep shows an elastic dominance (G' > G'') and no measurable relaxation time within the experimental time range. The creep test confirms elasticity with no steady viscous flow. The flow test reveals very clear shear-thinning behavior with viscosity dropping from $787 Pa \cdot s$ to less than $1 Pa \cdot s$. Overall the Mayo behaves like a viscoelastic solid with high viscosity at low shear rate and low viscosity at high shear rate.

4 Discussion

This study demonstrated the distinctions between Newtonian, Hookean and viscoelastic materials using bulk rheology techniques. Three distinct materials, agarose, glycerol and mayonnaise were used in this experiment. Oscillatory shear, creep and flow tests were done on each sample to provide an insight into the sample's elastic, viscous, and viscoelastic properties. Agarose demonstrated the behavior of a Hookean solid, with a dominant storage modulus (G') across all strain amplitudes and frequencies tested in Figure 2. The absence of a G'-G'' crossover indicates that elastic energy storage dominated over viscous dissipation throughout the test range in Figure 3. For the frequency sweep as shown in Figure 4, both G' and G'' were largely frequency-independent. The absence of a plateau in G' or measurable zero-shear viscosity supports the explanation that agarose behaves as a solid-like material with negligible viscous flow. The creep test in Figure 5 confirmed that under a constant applied stress, agarose exhibited minimal time-dependent strain and almost complete recovery upon stress removal. This behavior indicates an elastic solid with a very long relaxation time (τ) .

Glycerol exhibited classic Newtonian behavior, where viscosity remained constant across

all applied shear rates. The oscillatory tests showed G'' >> G' (Figure 6), indicating dominant viscous dissipation. The phase angle near 90° confirmed a nearly perfect viscous response and the linear viscoelastic regime extended across the entire range of strain amplitudes, reflecting the absence of a yield point or structural breakdown. The frequency sweep revealed that G'' increased linearly with frequency ($G'' \propto \omega \eta$), as predicted for Newtonian fluids and shown in Figure 7. Since glycerol molecules do not form cross-linked structures, stress relaxation is instantaneous relative to the oscillation period, yielding negligible elastic energy storage. The creep test provided further evidence of viscous flow. Strain increased linearly with time under constant stress in Figure 8, and the material showed no recovery after stress removal which is an ideal viscous response. The flow test produced a linear relationship between shear stress and shear rate, confirming that viscosity was independent of shear rate in Figure 9.

Mayonnaise displayed combining features of both solids and fluids. At small strains, G' > G'', indicating a soft solid material dominated by elastic interactions in Figure 11. As strain increases, G'' > G', signifying a fluid-like behavior. The G''-G'' crossover is characteristic of viscoelastic materials that transition from solid-like to fluid-like behavior beyond their yield point. However, our experimental data did not reach this crossover. This is most likely due to the experimental time being $\approx 30s$ (compared to the 60 s of glycerol and agarose). The frequency sweep showed that both G' and G'' were frequency-dependent in Figure 10, confirming time-dependent viscoelastic behavior. At low frequencies, viscous flow dominated (G'' > G'), while at high frequencies, elastic behavior (G' > G'') dominated. For the creep test, mayonnaise exhibited both instantaneous elastic deformation and a time-dependent strain increase in Figure 12. Upon stress removal, partial recovery occurred, indicating both reversible (elastic) and irreversible (viscous) deformation components. The flow test showed a shear-thinning trend, where viscosity decreased with increasing shear rate (Figure 13). Such non-Newtonian behavior is common in concentrated emulsions, gels and biological fluids.

This experiment highlighted how rheology bridges molecular interactions and macroscopic mechanical properties. The results of this experiment illustrate the fundamental rheological principle that real materials rarely behave as perfect solids or perfect fluids but rather as combinations of both, with behavior strongly dependent on structure, temperature and applied deformation rate.

5 Author Contributions

All authors contributed equally to this experiment and the write-up. N. Girard contributed to the data generation, background information and materials and methods. R. Tetteh contributed to the discussion. R. Lecuyer contributed to the experimental results and analysis section. M. Dunn contributed to the graph creation and analysis of experimental results.