Comments on Use of Reference Fluid to Verify DSR

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Baton Rouge, LA
September 16-17, 2014
Reference fluid – how and why?

- Used to verify torque transducer *ONLY*
- How can we do this?
  - Compare viscosity measured with the DSR to viscosity published for reference fluid
- Why can we do this?
  - Measure viscosity of reference fluid with DSR at combinations of *temperatures*/*frequencies*/*strains* where response of fluid is Newtonian
  - Newtonian $\Rightarrow$ viscosity is independent of shear rate
  - Elastic and viscoelastic response is therefore negligible and calculation for measured viscosity becomes straightforward – more later
Sidebar - how is viscosity of reference fluid determined?

- Reference values determined with capillary viscometer in region of Newtonian flow
  - Viscosity independent of shear rate
  - Shear rate is NOT given on reference fluid container

- When reference fluid is calibrated supplier must exercise same cautions used when DSR measurements are made
  - Stay in Newtonian (linear) region
  - Avoid shear thinning (non-Newtonian region)
  - In capillary experiment limits defined by temperature and shear rate
Viscosity measurement with DSR - two options

- Steady state shear
  - Apply constant uni-directional torque or shear rate until reach steady state flow
  - Issues with geometric linearity

- Better approach – use sinusoidal oscillation and assume resistance to deformation is due entirely to viscous flow
  - Perform at small strains where shear thinning is absent
  - Limiting strain that will give Newtonian flow depends on temperature and frequency
Items that might affect accuracy of DSR measurements using fluid

- Measurement temperature
  - Always verify DSR thermometer before using fluid

- Fluid expiration date
  - Do not use beyond expiration date – check label

- Heating the fluid
  - Heating can cause it to deteriorate
  - Use proper storage temperatures

- Improper test specimen preparation
  - Incorporation of bubbles can reduce measured value
  - Gap and improper bulge – good laboratory technique

- Each of above easily controlled with proper laboratory procedures
Determining steady state capillary viscosity from oscillatory shear in DSR

- Invoke Cox-Merz rule (1958)
  - Well known rule used for many fluids
  - Well accepted in literature
  - Equates complex viscosity to capillary viscosity

- At low frequencies (oscillatory) and shear rates (capillary) complex viscosity = capillary viscosity
  - Under these conditions elastic effects become negligible
    \[ |\eta^*(\omega)|_{\omega \to 0} = |\eta(t)|_{\partial \gamma / \partial t \to 0} \]
  - \( \eta^*(\omega) \) from DSR, \( \eta(t) \) from reference fluid bottle

- Note test conditions!
Measurement of Complex Viscosity, $\eta^*$

- Assuming only viscous response, $\eta^* = G^*/\omega$
  
  Substituting, $G^* = 2Th/4\pi r^4\Phi$
  
  $\eta^* = Th/2\pi r^4\Phi\omega$

where:

- $T$ = Torque applied to specimen, N-m
- $h$ = Specimen thickness, m
- $\Phi$ = Angular rotation, rad
- $r$ = radius of test specimen, m
- $\omega$ = frequency, rad/s

- If we assume other variables are accurately known, uncertainty lies with measurement of torque, $T$
Assumptions that allow T to be only variable of uncertainty

- Checklist
  1. Incorrect specimen geometry - diameter, gap, bulge ✓
  2. Improperly formed specimen - bulge, bubbles ✓
  3. DSR thermometer not verified - verify thermometer ✓
     - Fluid temperature susceptibility \( \approx \frac{1}{2} \) that of binder
  4. Angular displacement transducer ✓
  5. Internal instrument calibration ✓
  6. Machine compliance ✓
     - Minimal for strains at 64 and 70 C
- Above assumptions reasonable at small strains developed with fluid using 25 mm plate at 10 rad/s
- Remaining item is accuracy of torque transducer
Why test at single temperature?

- Measurement of question is torque
  - Torque transducer is at ambient temperature
  - No reason for testing at temperatures

- Torque at 64°C, 10 rad/s, 10% strain =
  - Similar to torque used with 8 and 4 mm

Rheometer Motor and Torque Transducer
Connecting Shaft
Fluid in Environmental Chamber
Viscosity – Reference vs. Measured as f(T)

- Recommend testing at 64°C
- Errors become large at lower temperatures
- Binder flows from plates at higher temperatures
Conclusion

- If DSR viscosity equals reference viscosity DSR is likely working correctly and torque transducer standardization is verified.
- If DSR viscosity differs from reference viscosity something is “wrong”
  - Could be torque transducer
  - Could be other internal DSR calibration items
  - Likely not machine compliance if strains are not small
  - Likely not temperature errors if temperature was verified
  - Likely not specimen issues if technician is competent