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Shear modulus of heavy oils: Confinement effects in rheometer measurements

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HIGHLIGHTS

- Heavy oils shear modulus increases when decreasing gap in parallel plate rheometers.
- We tested 3 samples using different gap thicknesses observing the same behavior.
- Heavy oils were tested at temperatures in which they behave solid-like $G' > G''$.
- This behavior is believed to be associated with the surface-active elements present.
- This finding can have major implications in flow of heavy oils in porous media.

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ABSTRACT

Viscosity of heavy oil constitutes an important property that governs the productivity of these reservoirs. The viscosity of these crude oils is highly dependent on many variables like temperature, frequency, and strain amplitude. In this paper we show that viscosity measurements of heavy oils can also depend on a measurement variable like the gap thickness in a parallel plate rheometer. In this paper we measured how the confinement of heavy oils between parallel plates induces an increase in shear modulus. We propose that the increase in shear modulus can be linked to the surface-active nature of heavy oils that can cause re-orientation of the molecules at the surface of the plates and producing a measurable increase in the shear modulus. The findings were observed in three different samples of heavy oils. The results of this work have major implications in the measurement of viscosity of heavy oils but more importantly in the flow of heavy oils in porous media.

1. Introduction

Heavy oils constitute the largest reserves of hydrocarbons in the world but still represent a small fraction of the production due to the difficult extraction processes involved. One major difficulty is the high viscosity of heavy oils at reservoir temperatures, often needing thermal Enhanced Oil Recovery (EOR) methods to reduce the viscosity in situ and improve productivity. Production and thermal recovery methods involve viscosity reduction based on its extreme dependence on temperature. Viscosity values can change orders of magnitude with relative small changes in temperature. Therefore the study of viscosity and shear modulus of heavy oils and their dependence on variables such as temperature, frequency and composition has been the center of many studies. One of the most common techniques to study shear properties of heavy oils is the rheometer, which is widely available and regularly used for these studies.

One important rheological characteristic of heavy oil is its viscoelasticity. Viscoelastic materials exhibit characteristics of both elastic solids and viscous fluids. An elastic solid is defined as a material that responds to Hooke's law [1], this means stress is proportional to strain. In contrast, a viscous fluid under shear stress responds linearly to the rate of strain and the constant of proportionality is the viscosity. Viscoelastic materials exhibit time or frequency dependence (also called dispersion) and require a function to describe its behavior. Frequency dependence of heavy oils has been measured by Batzle et al. [2], Han et al. [3], Spencer [4,5], among others.

Temperature dependence of heavy oils presents two regions where its properties change systematically, the glass transition

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and liquid point. At temperatures above the liquid point, heavy oils behave like viscous-liquids and below the glass transition they behave like solids. Between the liquid point and glass transitions heavy oil behaves like a viscoelastic material.

Temperature phase transitions between solid and liquid states, have been measured by several researches among them Han et al. [3] generalized the onset of the liquid and glass points for a heavy oil sample. Batzle et al. [2] also show the decrease of the shear modulus of a heavy oil sample when increasing temperature measuring changes in phase.

Similarly to the effect of temperature and frequency; composition plays an important role in the viscoelasticity of heavy oils. Heavy oils with large amounts of polar fractions (resins and asphaltenes) behave more like a solid, while heavy oils with large amounts on the lighters components (saturates and aromatics) behave more like liquids. Here we refer to asphaltenes, resins, aromatics and saturates as determines by SARA analysis. Indirect measurements of composition dependence have been observed by Hasan [6], Das [7] and Rojas [8] among others.

In this paper we show that another variable, in this case a measurement design variable, can induce phase changes in the heavy oil. We will show how the confinement of the heavy oil between the two solid–liquid interfaces in the rheometer can induce a transition from a liquid-like behavior to a solid-like behavior, similarly to the transition observed when reducing temperature, increasing frequency or changing composition. This phenomenon has been reported in other sciences as “solidification through confinement” or “liquid to solid transition due to confinement” but has not been extensively reported and explained in heavy oils at rheometer scales. Our results have important implications on measuring techniques of acoustic properties in geophysics applications and importantly in the study of heavy oil flow in porous media.

First, we will describe the materials, testing conditions and methodology of the experiments performed in this work. We then will show the results of the shear modulus versus gap thickness for three different samples of heavy oils. Next we will compare these results with similar observations found in the literature. Afterwards we propose a conceptual explanation for these results and finish the paper summarizing the implications and conclusions of this work.

2. Samples and testing conditions

Samples used in this research were obtained from three different outcrops in the United States. The samples are named based on the location and an internal laboratory code is used to preserve the information and data obtained. The three samples are named GP007-Uvalde, GP029-Asphalt Ridge and GP010-Goleta and Table 1 shows the available analysis of the samples and the location where each was collected. The three samples were extracted from the rock with toluene and the excess solvent was evaporated in the oven with occasional stirring to ensure a complete removal of the solvent. The complete removal of the solvent was observed by changes in volume of the sample with time. Evaporation temperatures were kept between 70 °C and 80 °C, since unpublished results in similar materials indicate that loss of mass can occur above 100 °C. These samples have different “gravities” or densities under standard conditions and viscosities at room conditions vary importantly. Therefore, testing temperature was varied from sample to sample to ensure they were in the viscoelastic regime and close to a solid-like behavior during testing. GP007-Uvalde was tested at 30 °C, GP029-Asphalt Ridge was tested at −6.5 °C and GP010-Goleta was tested at 40 °C.

Complex values of viscosity (η^*) were collected and these relate to the complex values of the shear modulus (G^*) through the following expressions:

$$\eta^* = \eta' + i\eta'' \tag{1}$$

$$G^* = G' + iG'' \tag{2}$$

$$\text{with} \quad G' = \eta''/\omega \tag{3}$$

$$G'' = \eta'/\omega \tag{4}$$

where η' and η'' are the real and imaginary components of viscosity respectively, G' is the ‘storage’ shear modulus and G'' is the ‘loss’ shear modulus and ω is the measurement frequency. Frequency sweeps were performed to all samples from 0.1 to 100 Hz. Strain amplitudes were kept between 0.02% and 0.04% to ensure testing was performed in the Linear Viscoelastic Regime (LVR) confirmed by strain sweeps. Gap thicknesses varied between samples as shown in the results, with larger gaps used for GP007-Uvalde and GP010-Goleta samples.

3. Methodology

Rheology experiments were performed in an ARG2 system from TA instruments, Inc. The rheometer has an upper rod connected to a parallel plate geometry and the lower piece is a Peltier plate which provides the temperature control. Calibration was performed daily as recommended by the manufacturer, some of the calibration steps included: rotational mapping, oscillatory mapping, inertia calibration and zero gap set-up. After the initial calibration, the sample is placed on the Peltier plate and temperature is raised to allow samples to melt but always kept below 80 °C. After the sample is melted, the geometry is lowered slowly until the largest desired gap plus few microns is reached. The sample should remain strictly between the geometry and the Peltier plate to ensure pure shear conditions, therefore the sample is trimmed carefully to remove any excess and the final gap is set. The temperature of the Peltier plate is set to the testing temperature and enough time is allowed for the sample to equilibrate. The temperature equilibration time for one of the samples (GP007-Uvalde) was calculated to be less than 20 min when the variations on the shear modulus is less than 1% with respect to the final stabilized value. Subsequently, the normal force is set to zero to ensure the sample is relaxed before performing the experiment. A frequency sweep is taken at the first gap. After this test is conducted, temperature is slightly elevated and a new reduced gap is set; the reduction of the gap can be done with no normal force

Table 1
Samples used with corresponding SARA and API gravity analysis[a].

Sample	Oil composition (fraction)				API gravity	Location
	Saturates	Aromatics	Resins	Asphaltenes		
GP007-Uvalde	0.02	0.26	0.24	0.48	−5.00	Texas
GP010-Goleta	0.23	0.25	0.25	0.47	NA	California
GP029-Asphalt Ridge	0.17 ± 0.01	0.31 ± 0.004	0.45 ± 0.01	0.07 ± 0.002	0.14 ± 0.01	Utah

[a] Standard deviation is provided when available.