PRF# 57692-ND9
Project Title: Altering wax appearance using shear and pressure
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Overview

Investigating wax appearance temperature under different shear, temperature, and pressure offers an opportunity to provide a more complete phase diagram and understanding of flow-induced phenomena in waxy oil systems, which is an important flow assurance problem. The students are engaged with hands-on laboratory experience using state of the art equipment, especially rheometry and scattering. The teamwork between graduate students, undergraduate student, and a visiting researcher allows for a unique opportunity for completing this research project. Research results are being compiled and will include an undergraduate honors thesis as well as M.S. and/or Ph.D. dissertations in addition to peer reviewed journal publications. At least 1 publication is expected in 2019.

The researchers are working collaboratively to compile four types of measurements in a comprehensive analysis of waxy mineral oil flow behavior. The first two measurements are rheological measurements at ambient pressure under static (no flow) and shear flow conditions. Static measurements measure phase change in a controlled temperature water bath. These results provide a control to compare with rheological tests at ambient pressure. Rheological testing includes flow sweep, peak hold, and temperature ramp tests. Select phase behavior and rheology findings are described below. The more difficult measurements include rheology under high pressure and rheo-small angle light scattering, which will be completed in the next year of funding. Once new insights are well documented, the project should allow the industry to construct wax phase diagrams with shear and pressure as independent variables without a full factorial experimental design.

Findings to date

Samples were prepared with various concentrations (wt%) paraffin wax in both light mineral oil (LMO) and heavy mineral oil (HMO) bought from Sigma Aldrich. Rheological measurements of waxy oil samples were conducted using TA Instruments DHR 3 rheometer. The experimental protocol uses a parallel plate geometry (60 mm diameter) at 0.5 mm gap to reach shear rates ranging from 1 to 1000 s^{-1}. Rheological measurements of pure LMO, HMO, and the pure wax serve as control measurements for waxy oil samples.

Phase change observations of six different wax percent samples in temperature-controlled water bath system should a significant variation in cloud point as wax concentration changes (Figure 1). All samples were placed in the water bath system at a temperature of 65°C while the temperature of the system was lowered at a different ramp rates of 1, 3, and 5°C/hour, to check repeatability of the phase changes.

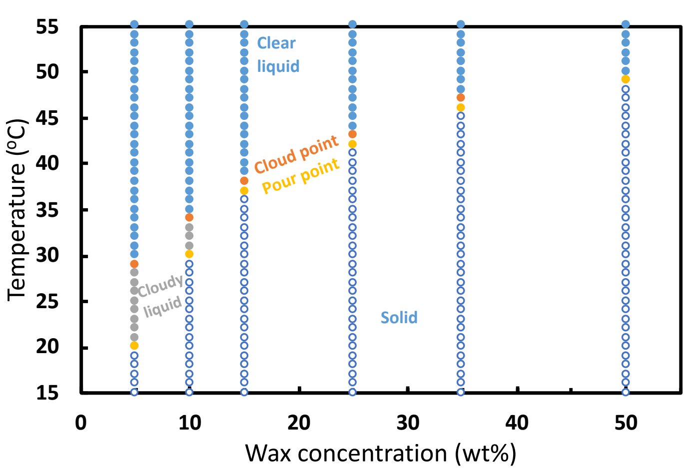

Figure 1: Static phase diagram

Visual observations quantify the phase of each sample. At 65°C, each sample starts as clear liquid, then gradually changes as temperature of the system is decreased. The changes observed ranges from appearance of a cloud (cloud point), viscous cloudy liquid, pour point, until sample finally turns to a white solid. Oil samples with low wax percent content exhibits a 2-5ºC gap between the cloud point (Wax Appearance Temperature) and the pour point; and this difference between cloud point and pour shrinks as the wax percentage increase until the 50% sample suddenly from liquid into a white solid, i.e., a convergence of cloud point and pour point.

The phase behavior at static conditions are being compared with phase changes under flow at 4 different shear rates. Flow sweeps measure viscosity at constant temperatures between 10 to 70°C and shear rates range of 1 to 1000 s^{-1}. Temperature ramps measure the phase change temperature at 4 different shear rates (1, 10, 100, 1000 s^{-1}) under a ramp rate of 0.5°C/min. Additionally, peak hold measurements maintaining a constant shear rate and temperature for 120 s followed by changing temperature in 0.5°C intervals. Initial findings show that shear alters the wax appearance temperature about 1°C.

Topics for second year

The current status of the project is at the beginning of the third stage. This includes small angle light scattering (SALS). The last stage of the project which includes rheological measurements at high pressure is also in progress. This involves saturating the samples with methane before introducing them into a high-pressure cell for rheological measurements to be repeated at pressures ranging from 0-2000psi. The complete high-pressure rheology system has been designed and implemented by graduate students in the lab (Figure 2). Leak checks and trial samples for an unrelated project were done and the system is ready for operation.

The unique experimental conditions are pressure and methane concentration, which are correlated. The working pressure range of the high-pressure rheometer system is 0 to 2,000 psig. This high-pressure system allows all the measurements of viscosity and phase change under flow. This high-pressure system provides a similar condition for the fluid which exhibits at the industrial plants and pipelines. The system consists of two main lines, gas supply line and the purge line connected to both a pressure cell rheometer and pressure reactor which will be working as the mixing and saturation chiller. High pressure pump will be used to transfer the sample under pressure from the reactor to the pressure cell of the rheometer.

A compilation of the research to date will be presented at the Society of Rheology (SOR) meeting on October 17, 2018 in Houston, which is critical to PI Liberatore's career. As the project continues to progress, papers will be prepared and submitted for publication in 2019.

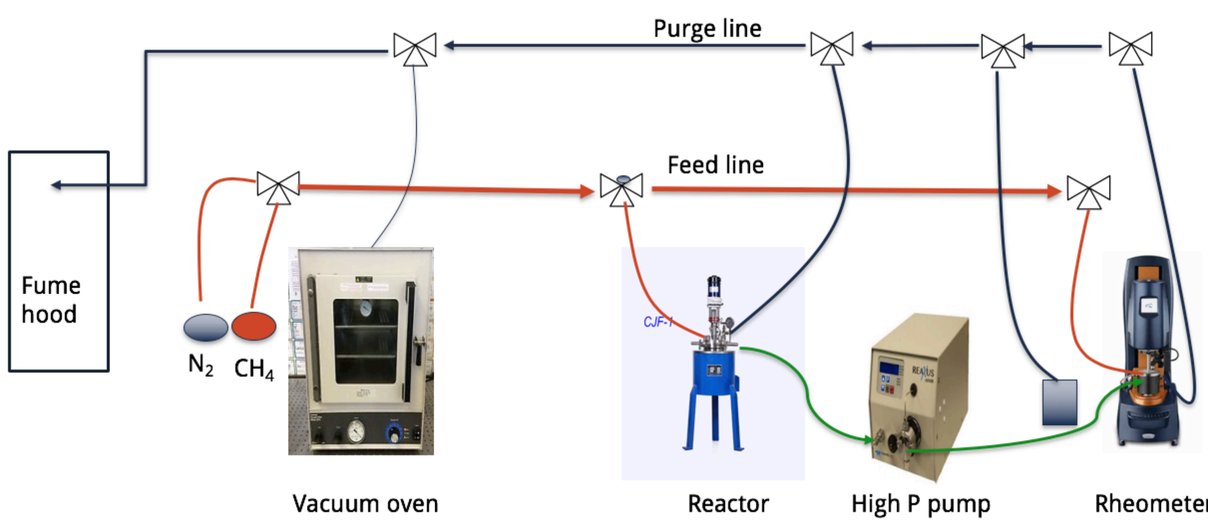

Figure 2: Diagram of high-pressure rheology system