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A practical method for the separation of high quality heavy oil and bitumen samples from oil reservoir cores for physical and chemical property determination

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HIGHLIGHTS

- Heavy oil recovered by mechanical compaction (plunger) is compared to centrifuge oil.
- Plunger oil is less viscous and contains less solid fines and water.
- Plunger technology can provide real time determination of viscosity profiles.
- The plunger can provide liter volume samples from core for PVT and other analyses.

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ABSTRACT

We describe a mechanical extraction method, referred to here as "the plunger", for the recovery of heavy oil and bitumen samples, equivalent to produced oil samples, from clastic and carbonate reservoir cores. We demonstrate the efficacy of the plunger relative to the centrifugation method through comparing the physical properties and chemical compositions of the heavy oils and bitumens recovered from oil sands cores. Over the dead oil viscosity range from 21,000 cP to 1.4×10^6 cP at 20 °C and 9.6×10^6 cP at 25.5 °C, the plunger consistently yielded correspondingly lower viscosity oils compared to the oils recovered by centrifugation from the same sample material, as well as lower sediment fines and water content. For an example of extremely viscous oil, the plunger yielded 3.3 g of 9.6×10^6 cP oil (25.5 °C), while centrifugation produced only 50 mg of fluid, adequate for geochemical analysis but insufficient for viscosity and density determination.

The plunger has many advantages that favor its use over centrifugation such as successful recovery of highly viscous oil from cores, lower oil sediment fines/water content and faster sample extraction (typically 30 min to 1 h versus 2 h). The plunger has also been operated at the rig site to generate oil viscosity logs immediately following core recovery (prior to or during petrophysical logging) affording real time data acquisition to support decisions for conducting production flow tests while drilling rigs are onsite. Incidentally, due to the improved preservation of physical properties controlling volatile liquid components, repeated plunging of larger volumes of sample core can be used to recover large enough volumes of heavy oil or bitumen for PVT or specialist assay analysis. Since the plunger is operated under a sealed system the device may be configured in such a way to interface with a PVT cell. Gas introduced into the plunger system ultimately can lead to the production and collection of "enlivened oils" for viscosity measurements.

1. Introduction

The "Plunger" is a sealed high pressure compaction system that compacts reservoir core at high stress, causing the oil to flow through a filter system to recover oil or bitumen from oil sand cores. The recovered oil is very similar to produced fluids [1,2]. The instrument functions essentially by mechanical compaction and works with reservoir cores (clastic or carbonate) and in some cases with very viscous oil in drill cutting samples. The most appropriate methods for the recovery of oil and bitumen samples from cores in preparation for viscosity measurements are designed to avoid contact of the oil with solvents and also encourage the retention of volatile components which have a major impact on the measured dead oil viscosity. Solvent extraction of oils from core is effective for geochemical needs but is inappropriate for physical

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property measurements. Firstly, it is difficult to ensure that the solvent has been completely removed from the bitumen which contributes to lower viscosities; and secondly, complete removal of the solvent also removes volatile components originally present in the oil leading to erroneously high viscosities [3]. Wallace et al. [4] introduced centrifugation as an alternative to solvent based extraction methods, although there were some drawbacks with the centrifuge process due to the presence of appreciable amounts of solids and water co-extracted with the oils. Recommendations proposed included that the water and solids should be removed; however, filtering was problematic with very viscous oils and the removal of water by sorption onto a column of dehydrating molecular sieve showed evidence for selective adsorption of bitumen components. Water may also be removed from the oil sample by distillation, although this procedure requires recombination of the lost volatiles, indicated by GC assay of the original oil sample. Furthermore, additional problems, including sample handling and processing invariably contribute to incidental evaporative loss of light end components from core [5] leading to progressively increasing viscosity values. The plunger provides an alternative mechanical method to centrifugation for the recovery of heavy oil and bitumen samples from reservoir cores that avoids these limitations.

The issues surrounding the reliability of heavy oil and bitumen viscosity measurements were investigated by Miller et al. [6]. In a double blind study, the viscosity of three identical samples varied by approximately ±10% for each lab and by ±40% among the labs, which was attributed to the different laboratory methodologies used for the preparation and measurement of viscosity of produced oils. Adams et al. [5] researched the potential causes of error associated with oil industry viscosity measurements and found even more significant variations among oils that were extracted from cores, while produced oils were also affected to a lesser extent. The primary controls on measured viscosity were identified as the storage conditions and length of storage time from core collection to oil recovery, along with contributions from water and solids content. Secondary effects on the viscosity data were assigned to the oil extraction method and temperature extrapolation method. These research efforts have led to closer attention being paid to the sample storage and processing protocols to minimize incidental loss of volatile liquid components leading to improved viscosity data amongst the heavy oil and bitumen industry.

The aim of this paper is to compare the physical properties and chemical composition data of a suite of heavy oil and bitumen samples that were recovered from clastic cores by centrifugation and the plunger. The samples originate from the Peace River oil sands area of northern Alberta, Canada and were chosen to span a wide range of physical properties with dead oil viscosities ranging from 21,000 cP to 1.4×10^6 cP at 20 °C, to 9.6×10^6 cP (25.5 °C).

2. Experimental

2.1. Samples

The selection of frozen stored core samples was based on viscosity data that were obtained previously from plunger recovered oils to provide the wide range in viscosity to test the efficacy of both plunger and centrifugation. The original intact core was sub-divided along the length of the core to provide comparable (side by side) samples to be submitted for oil recovery by the plunger and centrifugation. The centrifugation and plunging were carried out at the same time, and the dead oil viscosity measurements were performed within 1 day following mechanical extraction of the oils to enable direct comparison of the physical properties and geochemical compositions of the centrifuged oils versus the plunger recovered oils.

2.2. Mechanical extraction of bitumen from core samples

Bitumen is rapidly extracted from the core samples using the plunger. If frozen, the samples are left to thaw (soften) at ambient temperature for about 15 min. A 200–300 g sub-sample is selected from the core (ideally exceeding five weight percent oil/bitumen saturation) which is gently crushed using a hammer to thumb size to minimize volatile loss related to intensive sample preparation by crushing and grinding. Samples are sealed in the plunger cell and warmed to 60–80 °C in the closed cell prior to compaction, minimizing volatilization of light ends. Separate tests showed that the short duration (e.g. 1 h) heating process did not impact the rheology of the fluid in any way because the system is closed. The core sample is subjected to 5 tons of loading, gradually increasing up to 30 tons over 30 min. The oil is collected from the bottom of the plunger in a pre-weighed polypropylene container. Alternatively, if the system is pressurized with gas to generate enlivened oils (i.e. gas containing oils), then the produced oil is collected in a PVT cell. Following compaction, the oil samples are analyzed immediately upon collection to minimize loss of volatile components that could occur even during sample storage under refrigeration.

For centrifugation, the samples were prepared and loaded into centrifuge tubes. The centrifuge was operated at 40,000 rpm at 27–29 °C typically for 1.5 h. If the volume of fluid recovered during the spin cycle was <1 g the centrifuge process was repeated with additional fresh core material.

2.3. Low temperature viscosity measurements

Low temperature (range 10–80 °C) dead oil viscosity (cP) was measured using a Brookfield R/S – CPS + rheometer in the cone and plate configuration. The instrument was calibrated using standard fluids with known viscosity from 1060 cP to 5.6×10^6 cP (at 25 °C) with errors typically <5% (mainly 1–2%). Approximately 0.5 g of oil is loaded onto the viscometer plate and the appropriate spin speed (shear rate) is optimized through iterations achieving near 100% torque setting and the viscosity is recorded. The viscosity is measured over a temperature range 20–80 °C, and is recorded at each temperature using Brookfield Rheo2000 software (version 2.8).

2.4. Water content in oil by Karl Fischer analysis

The water content of bitumen samples (with no free visible water) was determined by using a Karl Fischer Titrator (Brinkmann–Metrohm Titrino Model 787 [7]). Water (weight percent) in the sample was determined by interpolation of a calibration curve determined by measuring the response of the apparatus to water standard samples, which were made with a known mass of water in a dry mixture of 26 vol% 2-propanol and 74 vol% toluene. The alcohol reacts with sulfur dioxide (SO_2) and base to form an intermediate alkylsulfite salt, which is then oxidized by iodine to an alkylsulfate salt. This oxidation reaction consumes water. Water and iodine are consumed in a 1:1 ratio and once all of the water present is consumed, the presence of excess iodine is detected voltametrically by the titrator's indicator electrode, which signals the end point of the titration. The resulting water content data typically falls within an error range of 3%.

2.5. Sediment fines content in oil

The sediment fines content in crude oil and bitumen samples is determined by extraction with toluene, following the guidelines in ASTM D473. The apparatus used is the Koehler Instrument Company sediment extractor which is composed of a 1000 ml flask, a condenser, a medium pore size (20–30 μm) extraction