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Comparative analysis of experimental methods for quantification of small amounts of oil in water



Konstantina Katika, Mehrdad Ahkami, Philip L. Fosbøl, Amalia Y. Halim, Alexander Shapiro, Kaj Thomsen, Ioannis Xiarchos, Ida L. Fabricius*

Technical University of Denmark, Denmark

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ABSTRACT

During core flooding experiments where water is injected into oil bearing core plugs, the produced fluids can be sampled in a fraction collector. When the core approaches residual oil saturation, the produced amount of oil is typically small (can be less than a few microliters) and the quantification of oil is then difficult. In this study, we compare four approaches to determine the volume of the collected oil fraction in core flooding effluents. The four methods are: Image analysis, UV/visible spectroscopy, liquid scintillation counting, and low-field nuclear magnetic resonance (NMR) spectrometry. The procedure followed to determine the oil fraction and a summary of advantages and disadvantages of each method are given. Our results show that all four methods are reproducible with high accuracy. The NMR method was capable of direct quantification of both oil and water fractions, without comparison to a pre-made standard curve. Image analysis, UV/visible spectroscopy, and liquid scintillation counting quantify only the oil fraction by comparing with a pre-made standard curve. The image analysis technique is reliable when more than 0.1 ml oil is present, whereas liquid scintillation counting performs well when less than 0.6 ml oil is present. Both UV/visible spectroscopy and NMR spectrometry produced high accuracy results in the entire studied range (0.006–1.1 ml). In terms of laboratory time, the liquid scintillation counting is the fastest and least user dependent, whereas the NMR spectrometry is the most time consuming.

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1. Introduction

Core flooding experiments are used for testing enhanced oil recovery strategies (EOR) on a laboratory scale. During these experiments, the oil-bearing core plugs are, for example, subjected to water injection. As a result, both oil and water are produced from the cores and typically collected in vials (Fig. 1). Quantification of the produced oil and water can be challenging, especially when the produced oil fractions are small, near residual oil saturation. A typical core flooding equipment (Fig. 1a) consists of: i) a piston displacement pump with fluid accumulators to force fluid into a core plug, ii) a test unit (the core plug inside a heated and pressurized core holder, to simulate oil reservoir conditions) and iii) a production unit, where the produced fluids are collected. Collection of the produced fluids (effluents) may be carried out by using a two or three-phase separator (Gupta et al., 2015). However, in this case, monitoring, for example, the presence of an active EOR component or changing pH and salinity is difficult (Stoll et al.,

2011). If continuous chemical analysis is required, the produced oil and water should be sampled in a fraction collector containing series of vials (Fig. 1b). Each vial collects a given amount of effluent during time and is exchanged for a new vial automatically.

In a typical core flooding experiment, the amount of oil in the produced fluid gradually decreases after water breakthrough. The recovered oil is distributed among several vials, and some vials may contain only a few microliters of oil. Due to capillary phenomena, the interface of oil to air is concave and the interface of oil to water is convex (Fig. 2a). The opaqueness of the oil will thus create an illusion of more oil within the sample than the actual volume. This causes a systematic error in visual measurement of the oil of about 200 μl higher than the actual volume. The recovered oil may also be disconnected from the continuous oil phase and be attached as film or droplets to the vial walls or form drops in the water (Fig. 2b) (Tang and Morrow, 1999). Quantification of this dispersed oil cannot be achieved simply by visual observations or weight-volume measurements.

In the present paper, we compare the determination of the produced oil by the different methods. The simplest method is based on directly reading high-definition photographs of the vials containing oil and water. The three other methods are based respectively on ultraviolet/visible (UV/visible) spectroscopy, low

* Correspondence to: DTU Byg - Department of Civil Engineering, Section for Geotechnics and Geology, Technical University of Denmark, DK-2800 Kgs. Lyngby, Denmark.

E-mail addresses: koka@byg.dtu.dk (K. Katika), ilfa@byg.dtu.dk (I.L. Fabricius).

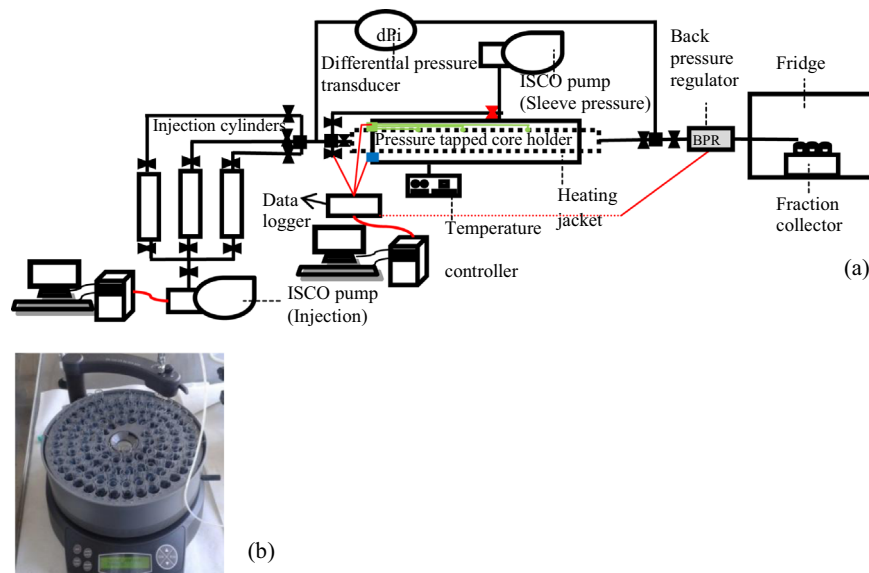


Fig. 1. (a) Core flooding apparatus and (b) fraction collector with vials.

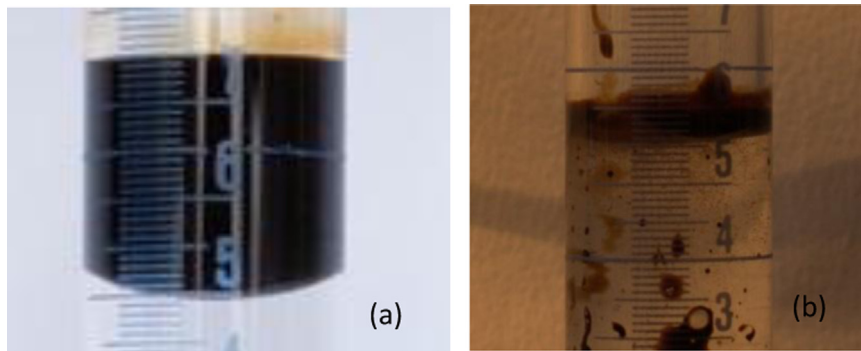


Fig. 2. (a) The curvature of oil menisci within the vials with 200 µl of oil and (b) dispersed oil on the walls of the vial.

field nuclear magnetic resonance spectrometry (NMR), and liquid scintillation counting (LSC). For each method we evaluated the range of applicability, the accuracy and the time resources involved.

2. Methods

2.1. Image analysis

Image analysis has been used for crude oil monitoring by several authors (e.g. Butler et al., 2001; Berg et al., 2010; Yang, 2011; Whitby et al., 2012). The method may be applied if the fluids are sampled in vials of identical geometry. Pal and Pal, 1993, discussed the main principle and operating procedure of the image analysis technique. According to their review, in high resolution images the estimated area is of high accuracy. A picture of each vial should be taken from the same position by a high resolution camera with a low distortion lens. Special precautions must be made in order to create uniform lighting and avoid reflections. The pictures are then analyzed with the help of digital analysis software for quantification of the oil volume. For the separation between near spherical oil droplets and more irregular solid particles, a shape factor defined as $4\pi \cdot \text{Area} / \text{Perimeter}^2$ will be one for spheres and less than one for other shapes. Gas bubbles can be recognized and excluded because they possess different optical properties from crude oil.

2.2. UV/Visible spectroscopy

UV/visible spectroscopy is applicable to quantify oil mixed in water when the oil contains asphaltene fractions, aromatic fractions or other functional groups absorbing light in the ultraviolet region (Bastow et al., 1997; Evdokimov et al., 2003a, 2003b). Evdokimov et al. (2003a), (2003b) and Yang (2011), discussed the standard operating procedures of this technique. Bastow et al. (1997) showed the reproducibility of the UV–vis method for quantifying oil in water. In addition, the authors also compared their results to the ASTM 3921 standard test method for Oil and Grease and Petroleum Hydrocarbons in Water and the results were consistent. Bastow et al. (1997), measured the absorbance at a wavelength $\lambda=256$ nm in order to obtain the total aromatic hydrocarbon content of a crude oil. One of the limitations of the method of Bastow et al. (1997), is that nitrate may interfere since it absorbs the wavelengths around 220 nm. Evdokimov et al. (2003a), investigated molecular aggregation of toluene solutions of crude oil and of a solid asphaltene. They used a pre-weighted Tatarstan crude oil and precipitated asphaltene from the oil and measured the adsorptivity at different wavelengths (λ). The samples showed similar absorbance at $\lambda=700\text{--}750$ nm. Shorter wavelengths ($\lambda < 550$ nm) give systematically higher absorptivity for solutions of crude oil in toluene as compared to pure asphaltene in toluene, due to other oil constituents (e.g. aromatics) (Evdokimov et al., 2003b). The principal approach of both studies is the same: oil samples are extracted with solvent and the absorbance is measured using a UV/Visible spectrophotometer. A standard curve

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