SEM IMAGING OF DRY AND SATURATED POROUS ROCKS FOR MODELING FLUID DISTRIBUTION ON PORE SCALE

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This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Calgary, Canada, 10-12 September, 2007

ABSTRACT

Scanning electron microscopy (SEM) is the only direct observational method that has a high enough resolution to allow for the investigation of details of the pore space morphology or very small objects like liquid films in the pores. We tested different preparation methods to obtain sedimentary rock samples that are suitable for high-resolution SEM imaging as a basis for a realistic description of the pore space. The produced photomicrographs were used directly in an applied modeling tool to simulate fluid distributions, e.g., at primary drainage.

To compare the model predictions with actual observations, sandstone and chalk plugs were saturated with synthetic formation water, and initial water saturation was established by injection of synthetic oil or crude oil at high temperature. The saturated samples were frozen in liquid nitrogen and investigated using SEM at low temperatures (Cryo-SEM) to obtain detailed information on the pore level fluid distribution.

Good results were achieved for saturated sandstone. The geometry of the wetting phase films attached to the pore walls and at the contact of the grains, as well as water-filled smaller pores, was clearly observed. The distribution of fluids could be fittingly simulated with our modeling tool. Due to the predominance of micro- and nanopores in chalk, the Cryo-SEM resolution impedes the distinction of fluids at high magnification in this rock type. The distribution of the fluid phases could only be clearly imaged in chalk macropores.

INTRODUCTION

In computation of effective multiphase flow properties in porous media and in understanding of underlying physical mechanisms, a realistic description of the microstructure is highly desirable. Scanning electron microscopy (SEM) is one direct observational method for generating realistic high-resolution images of the pore network. Even though SEM only provides images of the surface of a rock sample, it cannot be substituted by any other imaging tool in studying very small objects like liquid films in the pores and details of the pore space morphology because of high spatial resolution required. High-resolution SEM images of dry samples are often used for developing reliable 3D reconstructions of complex rock-pore systems and pore network modeling (Bakke and Øren 1997).

Recently established procedures for SEM at low temperatures (cryogenic scanning electron microscopy, Cryo-SEM) permit to analyze the in situ distribution of fluids in the pore network, offering unique information on fluid saturations and wetting properties (e.g., Robin et al, 1995; Boassen et al, 2006). The combination of Cryo-SEM images and X-ray elemental images allows for a comparison of network model predictions with the actual fluid distribution in saturated porous rocks.

EXPERIMENTAL

Well-studied and readily available outcrop rocks have been selected as standard material for the experiments: chalk from Liège, Belgium and sandstone from Bentheim, Germany.

Dry Sample Preparation

In order to be suitable as input to the network model, the SEM images need to be qualified for a digital separation of the sedimentary rock into a void and a solid phase. Different methods for the preparation of dry chalk and sandstone samples have been tested for their suitability to generate such images: fracturing, dry surface grinding, thin sectioning, as well as low-viscosity epoxy impregnation with subsequent rotary table surface grinding.

Saturated Sample Preparation

Core plugs are vacuum saturated with synthetic formation water, and drained to initial water saturation with synthetic oil or crude oil at high temperature by use of the single cell porous plate method. The core plugs are rapidly frozen in liquid nitrogen and cut into slices of about 4 mm thickness. The slices are stored in liquid nitrogen for further analysis.

Cryo-SEM Preparation

A frozen core plug slice is transferred to a steel box, which had been pre-cooled with liquid nitrogen. Small pieces of the sample are chopped off with a chisel and a hammer to fit a specially designed sample holder. The sample holder is attached to a transfer rod and cooled quickly by plunging into liquid nitrogen. The sample is transferred under vacuum to the preparation chamber, where it is fractured and sputter coated with platinum. After transferring to the SEM cold stage, it is observed at temperatures below -130 ºC.

SEM Imaging And Image Processing

The electron microscope used in this study is a ZEISS Supra 35 VP, equipped with an EDAX Genesis X-ray microanalysis system and a Polaron PP2000 cryo unit. The SEM is operated in high vacuum mode with an accelerating voltage of 12–20 kV, aperture size 30, and working distance of 8.5–11 mm. Best imaging results are achieved using the backscattered electron (BSE) detector in compositional mode. X-ray maps of C, O, and Ca/Si are generated to visualize the different phases in the saturated samples. ImageJ digital image processing software (http://rsb.info.nih.gov/ij/) is used for image enhancement (filtering) and feature extraction (thresholding).

Pore Network Model

The produced SEM images are used in a model for the distribution of two liquid phases in the void space of porous rocks at capillary equilibrium, described by Virnovsky et al (2006). The model consists of a bundle of capillary tubes of arbitrary crossections, obtained directly from the images and thus retaining some important features of realistic pore space geometry, like curvature and roughness of the pore walls. The capillary bundle geometry assumes that all the pores are accessible to the invading phase. Thus modeling of the oil-water interface inside the individual pores can be accurately done based on the SEM images; however, since the information on the spatial interconnectivity of the pores is missing, the total saturation picture can differ from the observations due to exaggerated accessibility of the pores.

RESULTS AND DISCUSSION

Dry Samples

The easiest and quickest preparation method for SEM imaging of dry sedimentary rocks is sample fracturing; however, the pore space is poorly defined by this method. The same applies to samples that are ground with abrasive paper to generate a plane surface. Standard thin sections have been found suitable for imaging chalk at low magnification only, or sandstone. The best results for both rock types for both low and high magnifications are achieved by applying a method based on two steps of low viscosity epoxy impregnation and subsequent rotary table surface grinding/polishing. With this method, the pore space as well as individual minerals can be distinguished from gray level values, and binary images of void (pore) and solid (matrix) can be generated (Figure 1).

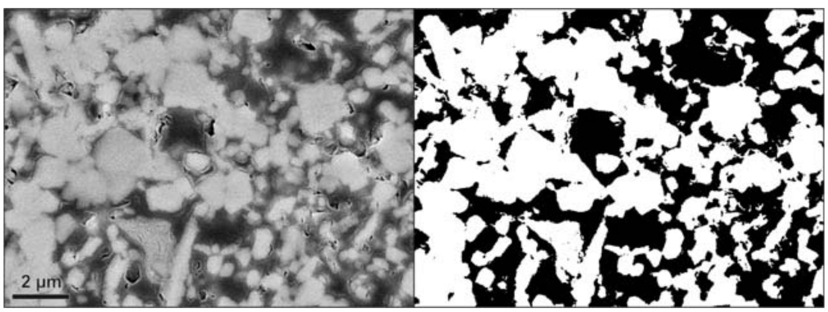

Figure 1. Left: High-resolution BSE image of chalk (left); dark gray is epoxy-filled pore space, light gray are grains. Right: Processed (filtered and segmented) image; white is solid, black is void; threshold level was set to the mean gray value.

With the applied modeling tool developed by Virnovsky et al (2006), drainage capillary pressure can be computed from suitable SEM images, and different saturations can be modeled. From comparing Figure 2 and Figure 3, which have similar magnifications, it is obvious that a high spatial resolution, i.e., a sufficient pixel density, is particularly important for modeling the narrow pore network found in chalk.

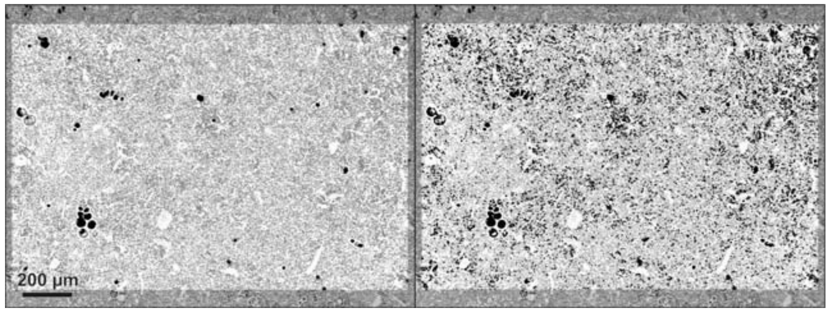

Figure 2. Low-resolution BSE image of chalk at two different simulated saturations. Image porosity is 38.99 %; oil/water interfacial tension, used to compute capillary pressure is 30 mN/m; area selected for simulation is in the center. In the simulation areas, white is matrix, black is oil and gray is water. Left: Water saturation 98.11 %, uncorrected *Pc* computed from image is 0.052342 bar. Right: Water saturation 67.67 %, uncorrected *Pc* computed from image is 0.126056 bar.

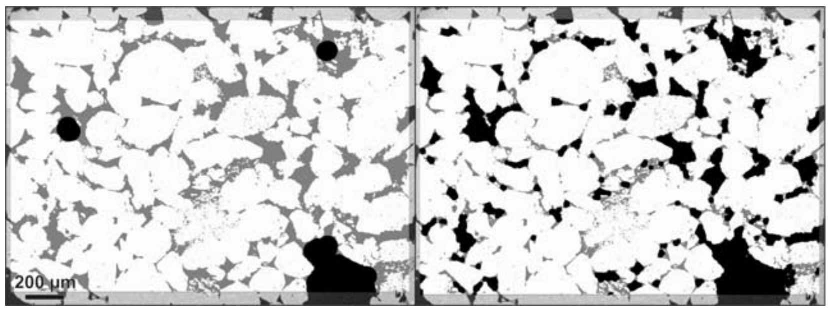

Figure 3. Low-resolution BSE image of sandstone at two different simulated saturations. Image porosity is 22.35 %; oil/water interfacial tension, used to compute capillary pressure is 30 mN/m; area selected for simulation is in the center. In the simulation areas, white is matrix, black is oil and gray is water. Left: Oil saturation 14.81 %, water saturation 85.19 %, uncorrected *Pc* computed from image is 0.005885 bar. Right: Oil saturation 67.76 %, water saturation 32.24 %, uncorrected *Pc* computed from image is 0.024231 bar.

Saturated Samples

With a combination of BSE images and X-ray elemental maps, fluid phases and minerals can be distinguished using Cryo-SEM (Boassen et al 2006). However, different problems arise for the studied rock types. Chalk mainly consists of tiny calcite grains, which results in a very narrow pore network. Too little fluid is present in these micro- and nanopores to be resolved; fluid distribution can only be clearly visualized in macropores (Figure 4).

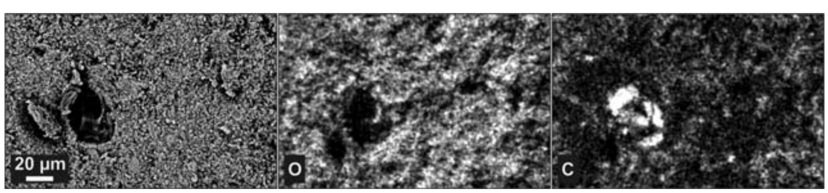

Figure 4. Chalk saturated with oil and brine. Left: BSE image; note macropore with dark gray infilling (oil). Middle: X-ray map of oxygen (white) showing the distribution of water; oxygen measured from grains had been removed. Right: X-ray map of carbon (white) showing the distribution of oil; carbon measured from grains had been removed; note high carbon intensities (accumulation of white pixels) also in smaller pores, indicating presence of oil outside macropores.

Good results have been achieved for sandstone, although sample preparation commonly results in too much topography, limiting the image quality. Nonetheless, the geometry of the wetting phase films attached to the pore walls and at the contact of the grains, as well as water-filled smaller pores, is clearly observed, see Figure 5. The detected fluid distribution in the pores can be directly compared with the model predictions (Figure 6). Analyzing the obtained SEM image we clearly observe that brine is present in the small pores, and also in the form of a film attached to the boundaries of the grains. The simulator basically captures the observed brine distribution in the smaller pores and oil in the larger, but does not capture the films. The reasons for this are believed to be due to:

(1) Incompleteness of 2D imaging of the 3D reality. An image only includes one curvature of a given surface, a pore wall or the oil/water interface. The films on the image may look exaggerated.

(2) The simulated fluids distribution is computed in 2D, i.e. the pore space is substituted by a bundle of capillaries with arbitrary crossections where the curvature of all the surfaces is zero in the direction orthogonal to the simulation plane.

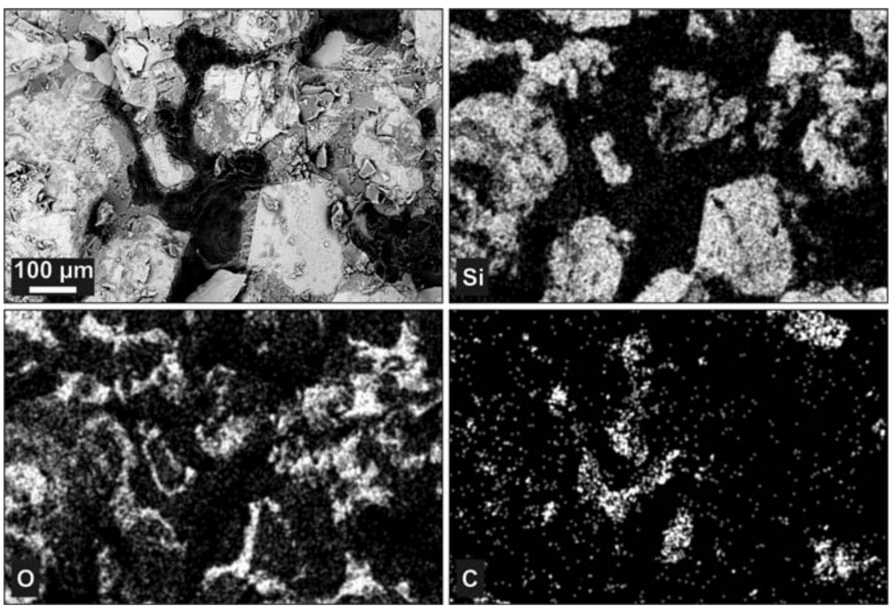

Figure 5. Sandstone saturated with oil and brine. Upper left: BSE image; light gray are grains, dark gray is oil, medium gray is brine. Upper right: X-ray map of silicon (white) showing the grains. Lower left: X-ray map of oxygen (white) showing the distribution of water; oxygen measured from grains had been removed. Lower right: X-ray map of carbon (white) showing the distribution of oil; carbon measured from grains had been removed. Note lower quality of X-ray elemental maps compared to BSE image, mainly as a result of topographic features.

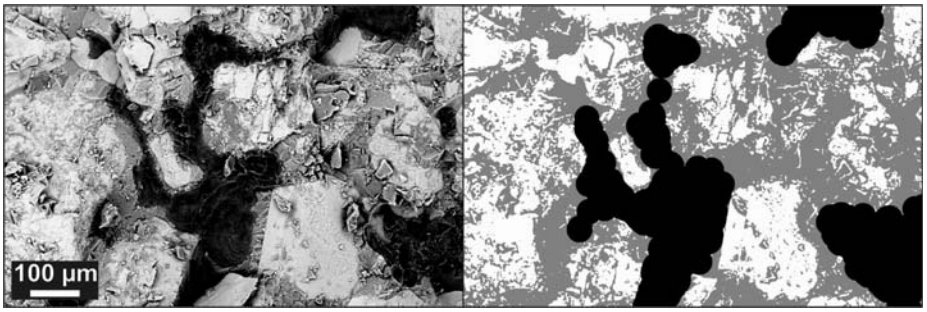

Figure 6. Sandstone saturated with oil and brine. Left: BSE image; light gray are grains, dark gray is oil, medium gray is brine. Right: Modeled fluid distribution; image porosity is 66.45 %; oil/water interfacial tension, used to compute capillary pressure is 30 mN/m; white is matrix, black is oil and gray is water; oil saturation 37.19 %, water saturation 62.81 %, uncorrected *Pc* computed from image is 0.040383 bar.

CONCLUSIONS

With a suitable preparation method, e.g., sophisticated epoxy impregnation and polishing, sedimentary rock samples can be prepared that are suitable for SEM imaging in order to be used for a realistic pore space description. After digital image enhancement and feature extraction, pore scale distribution of fluids can be successfully modeled directly from the images. Comparison of modeling results with in situ fluid distributions as observed with Cryo-SEM, allows for verifying and enhancing the pore network model. Cryo-SEM offers unique possibilities for observing and illustrating fluids in the pore network, notably in sandstone. Information on wetting properties, as well as fluid saturations can be obtained using this tool.

ACKNOWLEDGEMENTS

The authors acknowledge permission to publish the above paper containing results obtained within the COREC research project funded by Norwegian Research Council, and industrial sponsors ConocoPhillips Norge and Co-ventures, including TOTAL E&P Norge AS, ENI Norge AS, Norsk Hydro Produksjon AS, Statoil ASA and Petoro AS.

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