SMALL ANGLE NEUTRON SCATTERING ANALYSIS OF POROUS RESERVOIR ROCKS

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INTRODUCTION

Porous siliclastic sedimentary rocks are the most common reservoirs for oil and gas deposits. Interactions of fluids with the mineral surfaces in sedimentary rocks on the molecular scale have a crucial impact on hydrocarbon migration and on the possible exploitation degree of such reservoir rocks.

Typical reservoir simulation studies have to cover a size scale of tens of kilometers of lateral and vertical extend of the reservoir, while the relevant scale for fluid-mineral surface interactions reaches down to the nanometer scale. The mode of upscaling the nano-phenomena to reservoir dimensions is one side of the modeling problem. The other side is the rapid and efficient measurement of representative nanoscale parameters within a rock sample. Describing nanoscale surface features of large rocks is a difficult task, which requires the combination of various analytical methods as well as general concepts which can help to distill the relevant general quantities from huge data sets derived from individual specimen. For example, pore geometry can be described by shape factor attributes, which have to be measured for each pore individually either by a suitable imaging technique or a process-based model. Instead of trying to measure each type of pore and pore throat in order to deduce the parameters, a pore shape and throat system can also be described with fractal dimensions, based on the self similarity often found in these systems. Such a fractal parameter could describe the pore body and throat interaction in a simple and elegant form valid for a continuum of pores. In clastic rocks the fractal dimension describes clastic grains of different sizes, covered by various layers of cements, exhibiting a grainy structure on much smaller scales. This is a much simpler

case than for example the structure of a vuggy carbonate with fissures. Therefore we focus our investigation at first on sandstones.

Small angle neutron scattering (SANS) and ultra small angle neutron scattering (USANS) are such methods that promise to reveal insight in the self similarity of porous structures over a large scale, ranging from a few microns, a scale still accessible with conventional optical microscopy, down to nanometers, which come close to the molecular building blocks of the rock-forming minerals [1,2]. In this study we analyzed a variety of sandstone samples from a northern German gas deposit with SANS/USANS measurements. These sandstones exhibit a pore structure that was already described by fractal geometry to predict permeability. We compare the results to conventional core analysis and mercury injection data. Permeability in the Völkersen field is known to be very different for rocks of the same porosity. The reason is seen in the pore microstructure. From a descriptive view, a fractal model can account for this variability. The SANS and USANS data show a quantitative correlation between scattering vector and fractal dimensions of the pore structure.

Sample location

The Völkersen natural gas field is situated in lower Saxony between Hannover and Bremen at a depth of around 5000 m. It is composed of dominantly inter-dune and aeolian dune deposits of Permian age. It consists mainly of quartz grains as matrix constituent, feldspar content can reach up to 15%. Various rock fragments of crystalline and metamorphic or volcanic origin are rather common. Clay mineral particles (mainly illite with some chlorite) are present in very minor amounts, roughly between 3-7%. They are often present near the pore throats and thus may disturb the permeability even when present in traces only. Fe-oxide and hydroxide minerals were also observed as cements, next to sometimes strong quartz overgrowth. The sandstones have porosities around 12% with some regions reaching 18%. No gas flow is observed around 7% in-situ porosity. Permeability estimates from cores are around 20 mD average with regions of up to 2000 mD being present in the best dune facies. Permeability values reach down to about 0.01 mD, close to the tight gas boundary. The dynamic permeability as seen through production gas tests is lower, often 10 to 100 times below the interpolated and averaged core permeability.

Permeability is highly variable for a given porosity within the region. For a sample with a porosity of 12%, permeability can vary between 0.1 and 200 mD! The wide variation is caused by the different morphology and connectivity of the pore bodies and throats. The variability is even greater when comparing sandstones from different Rotliegend regions within Germany. The variability of the permeability is also observed in the variation of gas saturation and hence effective permeability. Low permeable rocks tend to show high irreducible water saturation and hence a low effective gas permeability.

Various methods for prediction and modeling of the permeability variations described here have been made in the past. Based on empirical fits to a huge database, H. Pape and others [3,4,5] established a fractal model to describe the permeability variations. They

computed the fractal parameter *D* from theoretical considerations. It has so far not been measured directly on the rock samples.

MATERIALS AN METHODS

The sample material comes from plugs cut out of cores taken from the reservoir. Unlike X-rays, neutrons are less absorbed in rocks and hence allow for an increase in sample volume. For SANS experiments, this benefit is countered by possible multiple incoherent scattering. The optimum thickness of the sample can hardly be calculated. We have prepared different rock samples of various thickness ranging from 0.5 to 4 mm. Cutting thin slices of rocks was difficult, and limited our material for the time being to Rotliegend samples that have sufficient cohesion. The samples had been cleaned and dried at 60 C. Routine porosity and permeability measurements were made on identical or twin plugs.

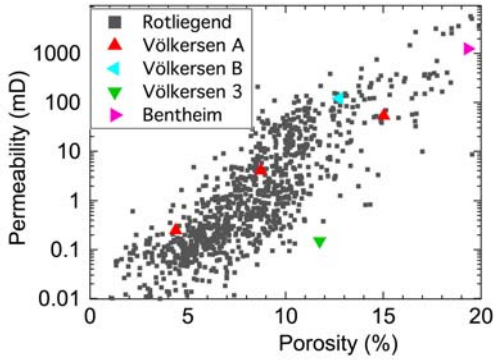

Figure 1. Comparison of porosity and permeability from Rotliegend and Bentheim sandstones (samples selected for the neutron scattering experiments are colored).

As the Rotliegend sandstone from the German Rotliegend is dominantly aeolian sediment with a long diagenetic history and a deep burial depth, it can be assumed that it represents a typical type of a mature sandstone, widespread in the Permian basins of Europe. As a tie-in to already published examples, a sample of commercially available Lower Cretaceous “Bentheim sandstone” was measured for comparison. This sandstone is widely known in Germany as dimension sandstone in construction industry but also has a good quality as aquifer and hydrocarbon reservoir rock. The selected samples have porosities and permeabilities that fit within the overall trend of the majority of Rotliegend samples (Fig.1), their parameters can be found in Table 1:

Sample	Porosity (%)	Permeability (mD)	Grain Density	Formation Factor	Description	Remark
2111	4.4	0.25	2.670	126	Rotliegend 1	Averaged from 2 plugs 10 and 60 cm apart
4146	8.7	4.04	2.656	43	Rotliegend 1	Averaged from 2 plugs 6 and 5 cm apart
5041	15.0	53.45	2.645	25	Rotliegend 1	Averaged from 2 plugs 12 and 15 cm apart
1460	12.7	120.92	2.638	33	Rotliegend 1b	
2440	11.7	0.15	2.656	68	Rotliegend 2	
2	19.3	1248.74	2.641		Bentheim	

Measurements were carried out at the Institut Laue-Langevin in Grenoble, France, on the instruments D11 (SANS) and S18 (USANS). A description of the Small Angle Neutron Scattering technique is given in [1,2]. Features responsible for neutron scattering can have a wide range of dimensions, from large pores with radii of 20 µm down to surface irregularities within the nanometer scale. The combination of SANS and USANS can cover this range to some degree. USANS has a typical resolution of 0.3 to 10 µm. Above 10 µm, multiple scattering events are dominant, even for samples as thin as 1 mm. SANS roughly covers the range of 0.3 to 0.003 µm.

RESULTS

Three samples from one Rotliegend well, having different porosities, were compared in an USANS experiment. The slope of Intensity *I* vs scattering vector *Q* was fitted with a power law, revealing the fractal dimension constant *D* [1]:

$$I(Q) = const \cdot Q^{-D} \tag{1}$$

It can be seen, that the samples show a decrease in *D* with increasing porosity and permeability. Samples from the two Rotliegend regions were compared to the Bentheim Sample and shows the difference in fractal dimensions (Fig. 2).

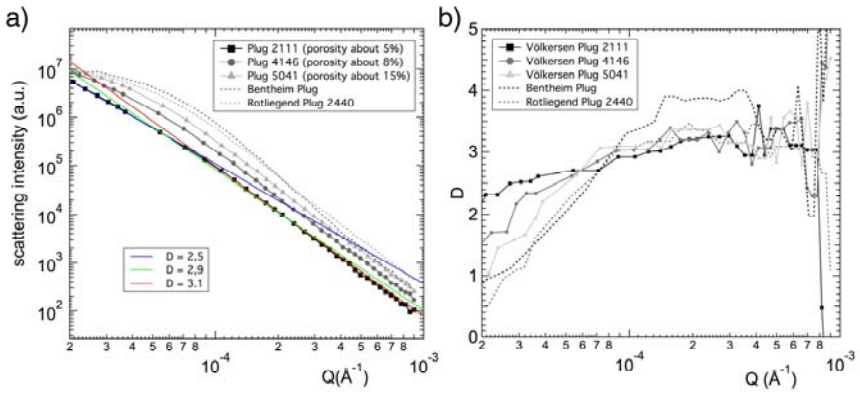

Figure 2. (a) Comparison between samples 2111, 4146 and 5041 (same well, porosity 5%-8%-15%) and another Rotliegend sandstone (different region, approx. 100 times lower permeability for similar porosity), as well as Bentheim sandstone. (b) Local slope of the USANS data sets (after moderate smoothing).

Different feature resolution reveals different fractal fitting parameters *D*. This can be seen by comparing USANS and SANS data over a wide range (Fig. 3). Large features point to a *D* around 2.2, while small features show a D value of up to 3.

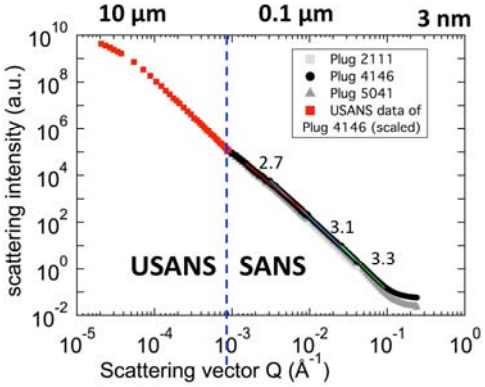

Figure 3. Comparison of SANS and USANS of Rotliegend samples. For sample 4146, fractal dimension D fits are made for various SANS resolution ranges.

INTERPRETATION

Using the formulas provided in [4], fractal dimensions can be calculated from rock samples, provided that porosity, permeability and formation factor have been measured. A comparison of the USANS derived fractal dimension values and the values calculated from petrophysical data show a very good agreement. The standard core analysis therefore shows features comparable to the USANS derived fractal dimensions (Fig. 4a).

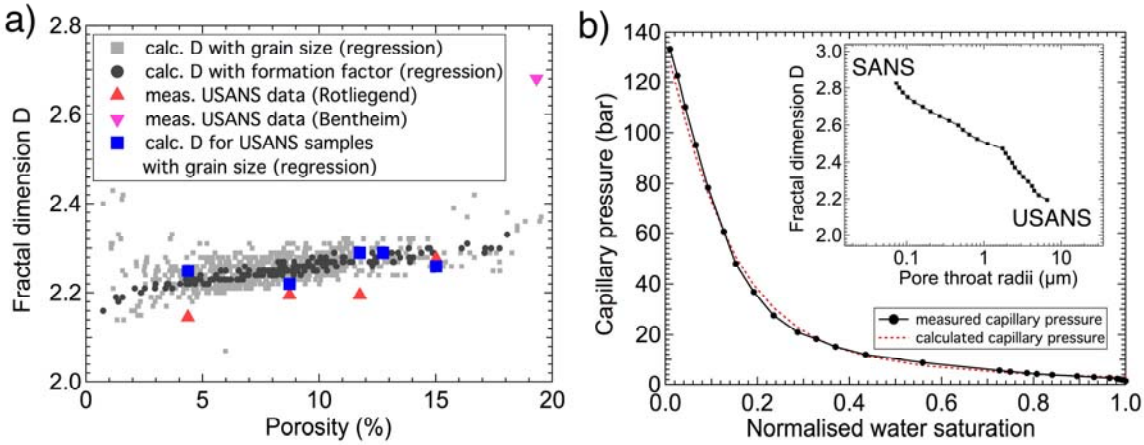

Figure 4. (a) Calculated fractal dimension from Rotliegend data (same as in Fig 1) using two methods, compared to experimental values from USANS. (b) Normalized mercury injection capillary pressure curve for sample 4146 (solid) and calculated from fractal parameter D (dashed line red), modified as shown in the inlay graph: Fractal dimension D calculated for each apparent pore radii calculated from the capillary pressure curves. Start and stop values are the D values from SANS and USANS fit.

Mercury capillary pressure curves are available for some of the samples investigated. Capillary pressure curves can be analyzed in terms of radii distribution and in terms of fractal dimensions [6]. Comparison of such an analysis demonstrates, that the low fractal dimension numbers derived from USANS are responsible for the capillary pressure behavior at lower pressures, whereas the behavior for the higher capillary pressures is only captured by the higher fractal dimensions as seen through the SANS experiment (Fig. 4b). The data suggest that surface effects are as important as pore radii at higher

capillary pressures. The radii inversion from capillary pressure data have therefore to be taken as an apparent value only that included also a roughness component.

CONCLUSIONS AND OUTLOOK

The SANS and USANS experiments reveal, that the fractal dimension number is a good descriptor of the pore scale features at various resolutions. They allow a description of formation factor, permeability and capillary pressure behavior without the need to image the rock directly through special µCT techniques. It is believed that this type of data will also improve the prediction of relative permeability curves where the traditional capillary bundle model often fails. More data and work are necessary to gain a better understanding of the mechanisms involved, this is particular true for the effect of wetting fluids in mineral surfaces. A correlation of imaged pore scale irregularities through AFM, laser scanning microscopy and other methods to the parameters obtains by SANS and USANS will allow a better correlation in future. This work is in progress [7].

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