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Comparison of the structure on the nanoscale of natural oil-bearing and synthetic rock

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Abstract

The structures of natural oil-bearing and synthetic rock were investigated using the ultra small-angle neutron scattering (USANS) technique. The scattering data from different thickness of rock were analysed using a Fourier transform method developed to remove the effects of multiple scattering and to simulate the scattering from a thin rock sample. It was found that only the oil-bearing rock could be analysed using this method. The synthetic rock samples could not be well described by this theory and this is interpreted as due to structural variation in the samples induced by the production method. All the rock samples are found to be surface fractals. The synthetic rock is found to have a much lower fractal dimension (D_s=2.0), indicating a smoother interface than the natural oil-bearing rock (D_s=2.7).
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Keywords: Oil-bearing rock; Synthetic rock; Ultra small-angle neutron scattering

1. Introduction

In the field of oil reservoir management, it is known that hydrocarbons are trapped in the pores of sedimentary rock and the efficient recovery is a matter of considerable concern to the oil industry. As much as 20% of the fluids in oil-bearing rocks can be trapped within pores of sizes of less than 1 μm. Clearly, it is advantageous to have a procedure to measure the porosity and pore size distribution at the nanometer scale before attempting to release these trapped fluids since the fluid transport processes will be related to the pore structure of the material (Tsakiroglou and Payatakes, 2000). The technique of ultra small-angle neutron scattering (USANS) was used to investigate the pore structure in sample reservoir rocks and to compare the results with those from synthetically produced rocks.

Synthetic rock is used to model reservoir processes (Petersen, 1997; Ismail, 2000; Manolas, 2002). For example, as the synthetic rock has a known and controllable composition, it is ideal for studying the effects of paramagnetic ions on the measured NMR signal, which can provide important information on the interaction of fluids with rock pores, for example. The effect of grain size on permeability of the rock to water and drilling mud can thus be studied in some detail. In comparison, the complex nature of natural rock can

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make the interpretation of lab-based experimental results quite difficult.

In previous studies, small-angle neutron scattering (SANS) has been used to study sedimentary rocks (Radlinski et al., 1996; Triolo et al., 2000) and coals (Senel et al., 2001; Prinz et al., 2004) over typical size ranges from 2 nm to 120 nm. In the present study, the USANS technique was used to extend the size range to approximately 10 μm. In both the SANS and USANS techniques, the intensity, I, of the scattering patterns have generally been found to follow a power law type behaviour with I dependent on the scattering vector, q, according to $I(q)=Aq^{-m}$ (where A is an instrument constant). The q is related to the scattering angle, 2θ, by $q=4\pi\sin\theta/\lambda$ where λ is the wavelength of the neutrons. The exponent m is usually a non-integer number between 3 and 4, which is interpreted as due primarily to a fractal surface between the rocks and the pores; however, since the void density is outside the dilute limit there will be a contribution from the inter-void correlations. The fractal dimension, D_s, is related to the power, m, by $D_s=6-m$, and a smooth surface will have $m=4$ and therefore $D_s=2$. A more complete mathematical description of the scattering from a fractal surface with a correlation length, R, and fractal dimension D_s (for $D_s<6$) is given by (Mildner and Hall, 1986)

$$I(q)\propto q^{-1}\Gamma(5-D_s)R^{5-D_s}[1+(qR)^2]^{(D_s-5)/2}\sin[(D_s-5)\tan^{-1}(qR)] \quad (1)$$

where $\Gamma(x)$ is the Gamma function for x. In this work, R is taken to indicate a maximum size of the pores in the rock. In the limit where $qR\gg 1$, Eq. (1) can be written as $I(q)\propto q^{-m}$.

Few studies have been undertaken to compare the scattering patterns of synthetic and natural oil-bearing rocks. Neutrons interact weakly with matter, with scattering length from different elements varying randomly with atomic number and with different isotopes of the same element (see, for example, Squires, 1997; Furrer, 1998). Small-angle scattering probes fluctuations in scattering length density (SLD) in a material. In the present study, the fluctuations in SLD are produced primarily by rock and void spaces and often the contrast can be altered by filling the accessible void spaces with solvent. Different isotopes of the same element have different scattering lengths; consequently, water and deuterated water have different SLDs. Therefore, a mixture of H_2O and D_2O can be prepared to have the same SLD as the rock matrix (equivalent to 50% D_2O), and in this case the mixture is said to be contrast matched to the rock. By filling the accessible space with the contrast matched mixture, the contrast between the rock and the filled voids is removed so scattering will not occur between the rock and the filled voids. Some pores will be inaccessible to the fluid and these 'closed' pores will still contribute to the intensity of the scattering pattern.

Whenever possible, samples for study by SANS and USANS are prepared so that neutrons undergo a single scattering event as they pass through the sample, thus avoiding an additional experimental correction to the data. Multiple scattering refers to the scattering of a neutron more than once as it passes through a sample. The degree to which experimental data from SANS and USANS measurements is affected by multiple scattering depends on the thickness, T, of the sample and the total coherent elastic scattering cross-section σ. The reciprocal of σ is the mean free path of the neutron within the sample, so that σT is the average number of times that a neutron undergoes a scattering event during its passage through the sample.

For practical reasons, many solid materials investigated using SANS/USANS techniques require the use of samples which are several millimeters thick (e.g., Itakura et al., 2005). This is particularly relevant for natural materials such as sedimentary rock where structural integrity and bulk properties (i.e., minimum surface effects) are important in sample preparation. Multiple scattering of the neutrons is expected in porous rocks (Radlinski et al., 2000a,b) and extracting the single scattering cross-sections from the measured data can be problematic. To do this, the value of σ needs to be found. The method used in the present study is the method of matching Fourier transforms, $s(r)$, of the scattered intensity, $I(q)$. This method requires measurement of SANS or USANS (preferably USANS) intensities from a number of samples of different thickness, at least two; however, three or four is better. For some initial value of σ, certain Fourier transforms are calculated from the data. The full details of the numerical modelling are found in (Sabine and Bertram, 1999). Briefly, the $I(q)$ from a sample of thickness, T, are fitted with the function

$$I(q)=A_0\sum_{n=1}^{\infty}\frac{(\sigma T)^n}{n!n^{\alpha}}\left(\frac{1+R^2q^2}{3n^{\alpha}}\right)^{-p}. \quad (2)$$

Here A_0, R and p are fitted parameters and α is derived from p, with p proportional to D_s. The value of σ (in this application the cross-section per unit volume, cm^2/cm^3) is adjusted over a suitable range (typically $0<\sigma<8$ cm^{-1}) until the Fourier transforms from the different thickness samples match (at least over some

part of the parameter range) and a value of R is obtained. From the fitted value of σ, the expected scattering intensity from a sample with no multiple scattering is derived. This is then fitted with the expected fractal scattering function shown in Eq. (1) to find D_s.

A major benefit of the technique of matching Fourier transform is that the method provides a check on the quality of the measured data. The data from different thickness samples must be consistent with kinematic multiple scattering theory (Schelten and Schmatz, 1980; Sabine and Bertram, 1999) and, importantly, the different data sets must be mutually consistent. A sample with a nominal thickness T must scatter like a sample of that thickness. If there are any variations in the sample or its surface, the method will show this as a mismatch of the Fourier transforms.

2. Experimental

2.1. Sample preparation

Oil-bearing rock was obtained from a drilling core from the Otway Basin off the south east coast of Australia. The core was sliced using a diamond saw into 5 and 8 mm thick slices. The core samples were left in their native state before measurement.

Synthetic rock was manufactured from unsieved silica grains with sizes of the order of 0.1 mm that were packed into a column of approximate diameter 35 mm and length of 100 mm. A proprietary activating solution was flushed through the column (Middleton and Kucharski, 2002) which replicates the process of natural calcite precipitation (calcite in situ precipitation system–CIPS). Briefly, flushing the solution through a sample precipitates calcium carbonate and the amount of calcite that forms between host grains can be adjusted by altering the number of flushes. This forms a porous mass representative of natural oil-bearing rock (Fig. 1) (Petersen, 1997; Ismail, 2000; Manolas, 2002). As with the natural oil-bearing rocks, samples were left in their as-prepared state and sliced with a diamond saw to the required thickness (4, 8 and 10 mm).

One set of samples was maintained in a dry condition; one set was soaked in D_2O in order to fill the available pores with a high-contrast, low-background material; and one set was soaked in a H_2O/D_2O mixture calculated to contrast match the rock material.

2.2. USANS experiments

The scattered intensity of neutrons incident on the sample was measured over a q range from 2.0×10^{-4} nm^{-1} to 1.0×10^{-1} nm^{-1} employing the perfect crystal diffractometer for ultra-high-resolution small-angle neutron scattering (USANS) instrument at the National Institute of Standards and Technology (NIST), Center for Neutron Research (Gaithersburg, USA). For the USANS measurement, the sample containers were placed in a two-position automated holder, and each q-scan measurement was accumulated for a total of 3 h. The neutron flux at the sample was approximately 1.7×10^4 n cm^{-2} s^{-1} at a wavelength of 0.238 nm. The full width at half maximum (fwhm) of the horizontal q resolution of the instrument is 2.5×10^{-4} nm^{-1} and the data were slit length smeared in the vertical direction with q-resolution of 1.1 nm^{-1}. Data reduction and background correction were made using NIST's USANS data reduction software.

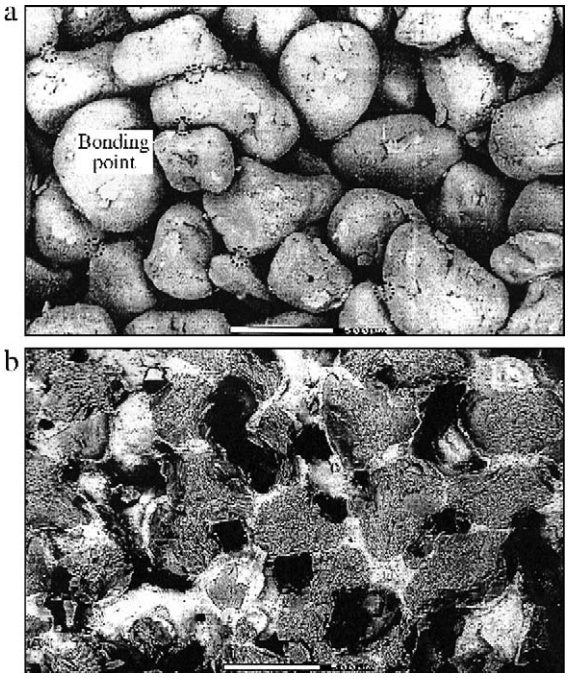

Fig. 1. SEM images of synthetic rock prepared under the following conditions: (a) one flush of CIPS through packed, unsieved silica grains, and (b) ten flushes of CIPS through the silica grains (Ismail, 2000). The light gray areas are calcite cement while dark gray areas are silica grains. Bar=500 μm.

3. Results

3.1. Dry samples

The USANS data from the dry oil-bearing rock are shown in Fig. 2 and their normalized ($s(r)=1$ at $r=0$ where r is the real space distance) Fourier transforms are

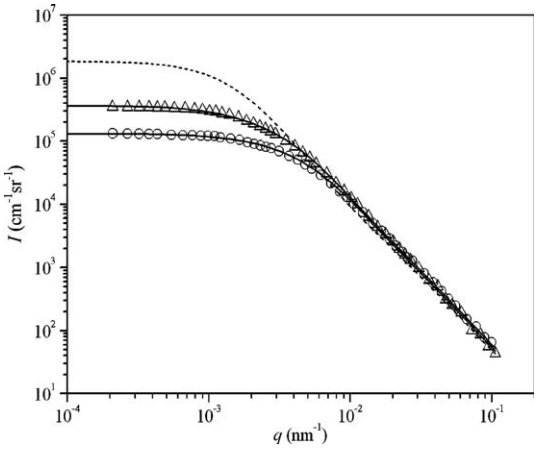

Fig. 2. USANS data from oil-bearing rock, 5 mm thick (triangles) and 8 mm thick (circles). Note that the experimental data are not slit desmeared and for clarity only every second data point is shown. The lines through the data are the fits to the data from the Fourier transform technique. The dashed line is the simulated scattering intensity of an infinitely thin sample.

shown in Fig. 3. The close matching of the transforms over a large range of r-values in Fig. 3(b) indicates that the value of the total elastic scattering cross-section (per unit volume) is $\sigma = 6.5$ cm^{-1}. This value was then used to calculate the single-scattering intensity. Using a fractal model for the rock it was found that the surface fractal dimension is $D_s = 2.7$ and the correlation length is $R = 660$ nm. The scattering curve corrected for multiple scattering is shown as the dashed line in Fig. 2.

The analysis of the data from the natural oil-bearing rock samples shows that the scattering from these samples is consistent with multiple scattering theory. Carrying out the same analysis on the data from the synthetic rock was not so successful. The experimental data are shown in Fig. 4 and the normalised Fourier transform data in Fig. 5. As seen from Fig. 5, even when σ is taken as high as $\sigma = 8$ cm^{-1}, it is impossible to obtain matching Fourier transforms.

In order to verify these results, the USANS experiments were repeated on a separate occasion with a slightly different instrument configuration (slit size and sample-detector distance) and with samples cut from the same billet (three thicknesses 4 mm, 8 mm and 10 mm). The results are shown in Figs. 6 and 7 and are, within experimental error, the same as Figs. 4 and 5.

The data from the synthetic rock samples are not consistent with kinematic multiple scattering theory. Although it is possible that, for this material, the theory is inadequate, it more likely that one or both of the samples are not homogeneous on the length scale examined by the USANS technique.

Further evidence for differences between the natural oil-bearing and synthetic rock comes from the fitting of a power law model to the desmeared data for the synthetic and oil-bearing rock. The 5 mm thick oil-bearing rock was fitted with a power of -3.3 ± 0.1 ($D_s = 2.7$) while the 8 mm thickness was fitted with a power of -3.2 ± 0.1 ($D_s = 2.8$). The 4 mm and 8 mm thick synthetic rock samples were fitted with a power of -3.9 ± 0.1 ($D_s = 2.1$) and -4.1 ± 0.1 ($D_s = 1.9$), respectively. These values of fitted powers are close to -4 as would be expected for a completely smooth surface, indicating that the fractal nature of the natural oil-bearing rock is not well replicated by the synthetic rock, at least on the length scale probed by USANS.

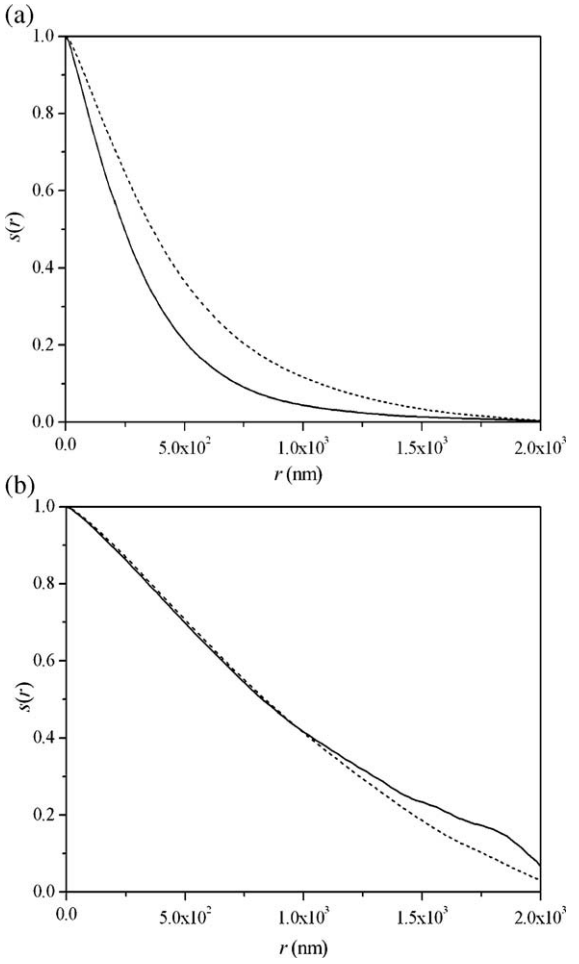

Fig. 3. Normalised Fourier transforms of USANS data (Fig. 2) from 5 mm (dashed line) and 8 mm (joined line) thick natural oil-bearing rock calculated with (a) in the limit $\sigma \rightarrow 0$ cm^{-1} and (b) $\sigma = 6.5$ cm^{-1}.

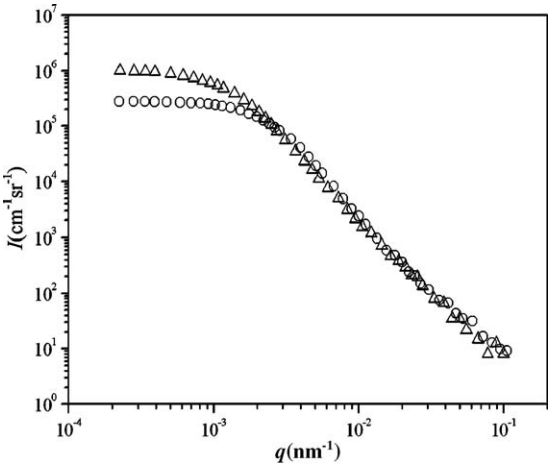

Fig. 4. USANS data from synthetic rock with 4 mm (triangles) and 8 mm (circles) thickness—Series I experiments. Note that the experimental data are not slit desmeared and for clarity only every second data point is shown.

3.2. Wet samples—contrast matched

The USANS data for both the natural and the synthetic rocks, where the samples were soaked in a mixture of light and heavy water to contrast match with the rock, are shown in Fig. 8. The method of matching Fourier transforms failed to produce satisfactory results for both samples. To check the effect of contrast matching, the desmeared experimental I from (i) the three fully deuterated samples and (ii) the corresponding partially deuterated samples, were integrated over scattering angle 2θ to determine σ (not corrected for multiple scattering). Contrast matching should greatly reduce σ; however, examination of the results in Table 1 shows only a small effect, which indicates that contrast matching had not been achieved throughout the sample.

In Fig. 8(b), the scattering curves corresponding to the two different thickness of the synthetic rock clearly do not overlap at large q as expected. In particular, the scattering profile above 3×10^{-3} nm^{-1} of the 4 mm thick sample does not display the expected power law dependence.

4. Discussion

Of all the data examined, only one set (that from the dry natural oil-bearing rock) is fully consistent with the theory of multiple scattering. The data from the natural oil-bearing rock permeated with deuterated water was only partially consistent with theory. The USANS results from the oil-bearing rock samples are more straightforward to explain and are consistent with the results of other studies on sedimentary rock (e.g., Radlinski et al., 1996; Triolo et al., 2000). The fact that the Fourier transforms of the dry material were in excellent agreement indicates that material in those samples is uniform throughout the sample. It is recognized that cutting the material into thin slices may cause a change in the number or size of pores close to the surface by relieving local stresses or filling of surface pores with fine fragments of waste material. This effect would be expected to be more pronounced in the thinner samples; however, there is little difference between the different thickness of the dry natural oil-bearing rock. It was therefore concluded that the differences observed between the dry and deuterated oil-bearing rock samples are probably due to the deuteration process. Deuteration relies on the mechanisms of diffusion and ion exchange, which can take a

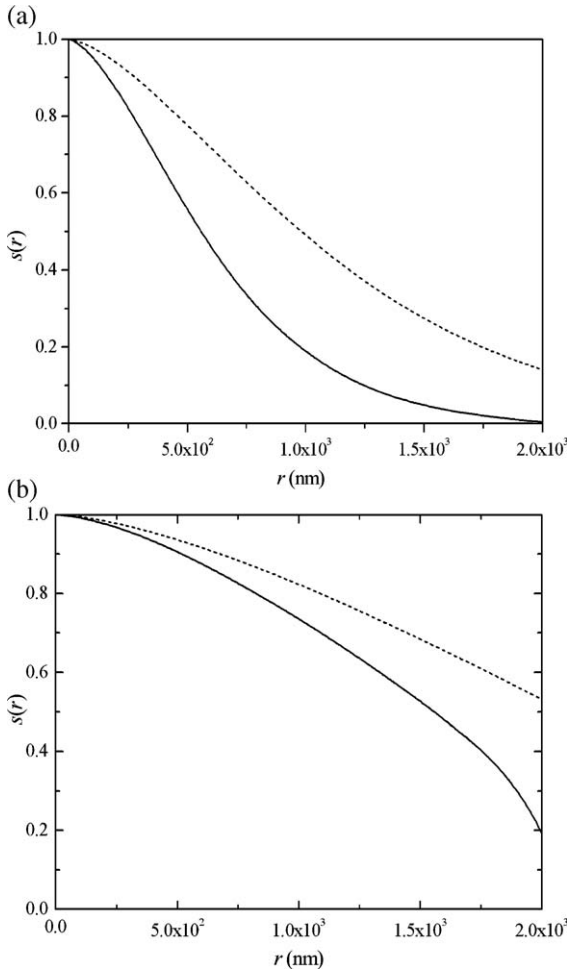

Fig. 5. Normalised Fourier transforms of USANS data (Fig. 4) from 4 mm (dashed line) and 8 mm (solid line) thick synthetic rock calculated with (a) in the limit $\sigma \rightarrow 0$ cm^{-1} and (b) $\sigma = 8$ cm^{-1}.

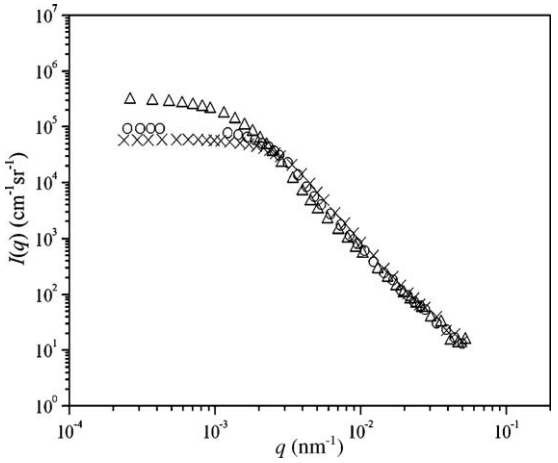

Fig. 6. USANS data from synthetic rock, 4 mm thick (triangles), 8 mm thick (circles) and 10 mm thick (crosses)—Series II experiments. Note that the experimental data are not slit desmeared. The section of data missing from the 8 mm thick sample was due to an error in setting up the scan sequence.

long time to complete. Whilst sedimentary rock should contain very little closed porosity, it is conceivable that some porosity will be inaccessible by a passive diffusion process (expect on very long time scales). Therefore, in this case, the filling of the pore space of the samples with water was not completed, which also explains why there was very little difference between the measurements from the fully deuterated and the contrast matched samples.

The general lack of agreement in the Fourier transforms for the synthetic rock presents a challenge. The synthetic rock samples appeared not to be uniform throughout at least on this length scale. The 4 mm thick sample does not show the typical power law decay expected with this material and it was not possible to find agreement between the Fourier transforms of the two (Series I) and three (Series II) different thickness samples. These differences may be due to an inherent characteristic of the synthetic rock or to the preparation method used to slice the samples.

One possible explanation for the discrepancies of the Fourier transform from synthetic rock of different thickness is that there is significant difference between surface layers and the bulk material. However, the synthetic rock samples were sectioned using the same method as the natural oil-bearing rock which showed no such difference. The effect may be associated with the production of the synthetic rock material using CIPS. The synthetic rock is originally produced in a cylindrical shape approximately 100 mm long and the slices are then cut from the cylinder. If the synthetic rock is not uniform along its long axis, or indeed across a cross-section, then the slices of material which will be taken from different points along the axis will not have the same scattering pattern. Previous electron microscopy evidence (Manolas, 2002) and measurement of the pore size suggest that the pore size distribution of the synthetic rock is reasonably uniform along the long axis. Some caution is necessary in a direct comparison of results. Image analysis techniques were used to interpret the SEM images (typically an area of 5 mm^2) and derive pore sizes; however, in a USANS experiment, the correlation in positions of voids and the roughness of the interface between the void space and the silica structure are being probed over a considerable volume (typically $\sim 10^3$ mm^3 depending on sample thickness). Hence, while the pore volume may be similar, the distribution in position of voids and structural features of the interface may change along

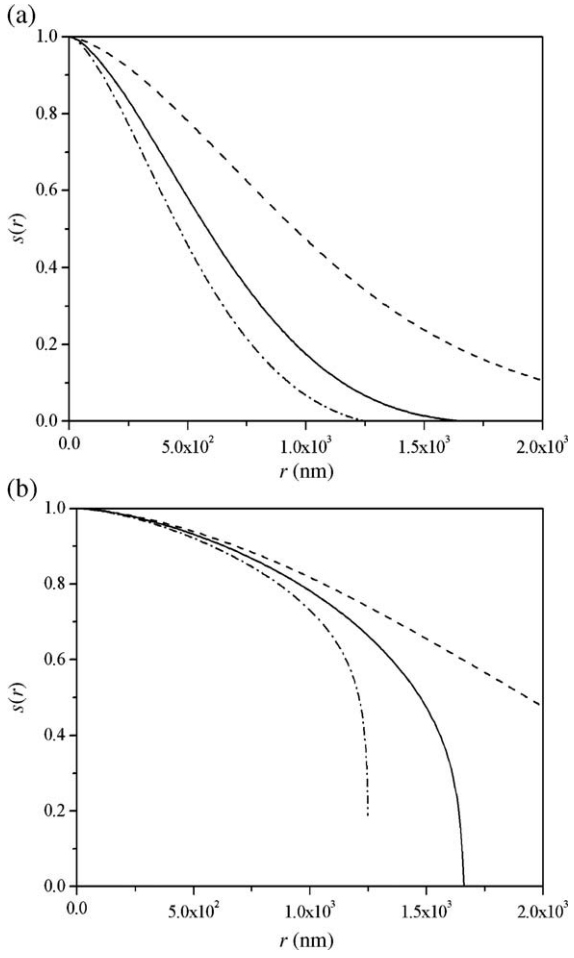

Fig. 7. Normalised Fourier transforms of USANS data (Fig. 6) from 4 mm (dashed line), 8 mm (solid line) and 10 mm (dot-dashed line) thick synthetic rock calculated with (a) in the limit $\sigma \rightarrow 0$ cm^{-1} and (b) $\sigma = 8.0$ cm^{-1}.

the axis of the cylinder. For example, this may be caused by variation in the rate of flow of the activating solution through different regions of the cylinder during the CIPS production of the synthetic rock. Further studies are required to explore the possible reasons for the observed differences in the different thickness of material, particularly given the reproducibility between Series I and Series II USANS experiments.

Further evidence of the differences between the scattering nature of the synthetic and natural oil-bearing rocks is found in the fractal nature of the samples. The natural oil-bearing rocks have a surface fractal D_s of 2.7. This indicates a surface with self-similar features over several orders of magnitude in size and is similar to that found in previous studies on sedimentary rocks (Radlinski et al., 1999; Radlinski et al., 2000a,b). In comparison, the synthetic rock has a fractal dimension close to 2 implying the surface of the structure is relatively smooth. The proprietary activating agent used in the production of the rock may contribute to its smooth surface by dissolving a thin surface layer of the silica grains and so artificially smoothing the interfacial surface between the silica and the pore space. Crampin (1999) outlined the necessity of considering the fractal nature of rocks when investigating or modelling reservoir behaviour.

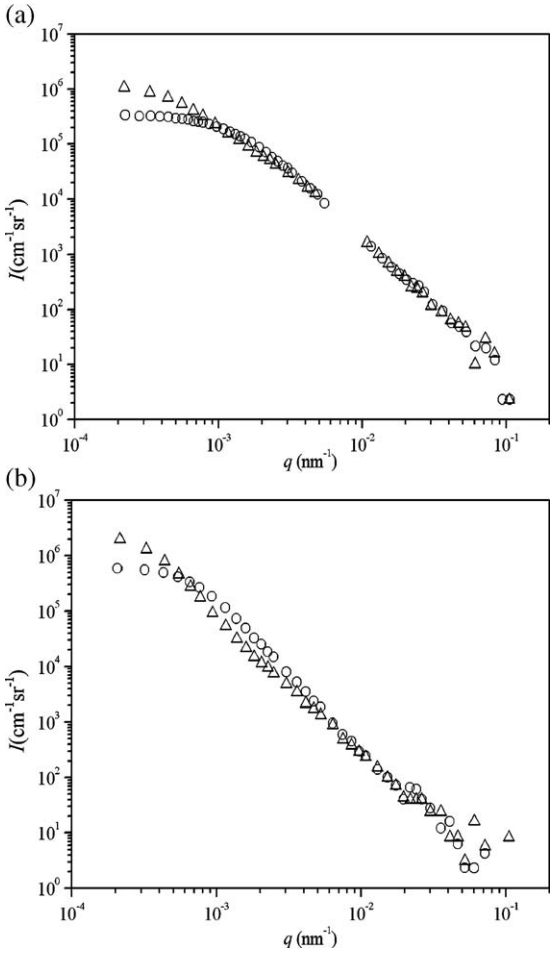

Fig. 8. USANS data from the contrast matched (a) oil-bearing rock in 5 mm (triangles) and 8 mm (circles) thickness and (b) synthetic rocks in 4 mm (triangles) and 8 mm (circles) thickness. Note that the experimental data are not slit desmeared.

Table 1
The desmeared integrated differential cross-sections per unit volume from three fully deuterated samples and the corresponding partially deuterated samples

Rock	Thickness (mm)	Cross-section per unit volume (cm^{-1}) 100% D_2O	Cross-section per unit volume (cm^{-1}) 50% D_2O
Oil-bearing	5	1.98	1.66
Oil-bearing	8	1.30	1.14
Synthetic	8	1.39	1.04

The cross-sections σ have not been corrected for multiple scattering.

5. Conclusions

Multiple scattering theory was used to interpret the USANS data from natural oil-bearing and synthetic rock. The theory described the natural oil-bearing rock well, however, did not describe the scattering from the synthetic rock. This is attributed to inherent characteristics of the synthetic rock. The fractal nature of the oil-bearing rock is in agreement with previous studies of these types of materials with the rocks having a surface fractal with $D_s = 2.7$. The synthetic rock does not display the same fractal behaviour having a fractal dimension close to 2 indicating a smooth surface. The difference between the scattering properties of the rocks seems to indicate that the synthetic rocks are not good substitutes for natural oil-bearing rocks in USANS experiment. This may have implications for the use of synthetic rocks in other experiments.

The matching Fourier transforms method of analysing small-angle scattered data (both SANS and USANS) is widely applicable and particularly for porous rocks with fractal structure.

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