PORE SIZE CHARACTERIZATION OF VUGGY CARBONATE MATERIAL

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ABSTRACT

Vuggy carbonate material pose significant challenges when it comes to characterization at the pore scale level. The material under study is a highly porous and connected vuggy system with vugs in the millimetre range and permeabilities correspondingly in the tens of Darcy range. Depending on vug accessibility and connectivity, pore size distributions from capillary pressure based methods such as mercury capillary injection curves can greatly underestimate the larger elements of the actual pore size distribution, even when viewed as a pore throat distribution.

Comparison of results from capillary pressure curves and pore size distributions obtained through a combination of NMR relaxation and diffusion measurements show that the mercury capillary pressure curves, are in fact, quite insensitive to the vuggy portion of the pore space and mainly reflect the properties of matrix porosity. The NMR data clearly show a partitioning between vuggy and matrix porosity, and thus comprise a valuable tool for pore size characterization for such materials.

INTRODUCTION

Carbonate formations comprise more than fifty per cent of the worlds recognized petroleum reserves. Compared to sandstones, carbonate reservoirs show a larger variety when it comes to petrophysical properties both on the core scale level as well as on the reservoir scale level. In turn this makes measurements and subsequent interpretation of special core analysis data more challenging than for similar sandstone cases.

One of the issues with the larger heterogeneity of carbonate samples is that the representative elementary volume to a large extent relies on the property of interest as well as the method of measurement. This is in particular true for dual porosity systems such as vuggy materials. Vuggy facies make up a significant part of the carbonate reserves, meaning that a better characterization of such is of great importance. Vuggy systems are often divided into two subcategories, labelled as separate and touching vugs respectively. The categorization depends on how the vugs are connected. In the first case vugs are thought to communicate only through the matrix whereas in the latter case the vugs can form a continuum throughout the media.

To investigate such a material in terms of pore size characteristics it is useful to explore different methods for determining the internal structure as all the methods have their limitations in terms of regions and properties they represent. It is therefore also important to gain knowledge of where the limitations of each technique affect the significance of the interpretation of the measurement.

The standard method for obtaining pore size distributions in porous media is through mercury capillary curves. (Dullien 1992) Mercury injection is a percolative pore size measure as the capillary pressure curves are interpreted as a pore size distribution by regarding the porous media as a bundle of cylindrical tubes, and then assigning the volume invaded at a given mercury pressure to the volume of pores with radius corresponding to the given pressure. As porous media hardly ever actually consist of cylindrical tubes, the mercury curves in practice reflect a pore throat distribution. In addition, due to the percolative nature of the method, where mercury is injected from the outside of the sample, smaller pore sizes will be overestimated as the volumes of large pores accessed through a smaller pore will be assigned to the smaller pore. In a fairly homogenous system, the results from a mercury capillary pressure curve reflects the true structure to a satisfactorily degree. However, in more heterogeneous systems where connectivity and accessibility can vary significantly, interpretation becomes more uncertain.

NMR has significant advantages over the mercury capillary pressure curves. Contrary to mercury based measurements, NMR is a tool that is non-destructive to the sample. This means that there is no need to sacrifice samples in order to obtain information on the structure of the rock. The NMR signal also measure the entire sample as whole, implying that there is no distinction between pore throats and pore bodies, and the resulting distribution can be interpreted as a pore size distribution through the relation by Brownstein and Tarr (1979), where ρ is the surface relaxivity of the given sample.

$$\left(\frac{1}{T_2}\right) = \rho\left(\frac{S}{V}\right) \tag{1}$$

The relation also shows that pore size measurements by NMR has the advantage that there is no model imposed on the distribution, but that the data merely reflects the surface to volume ratio of the sample. The problem of using NMR as a pore size measure has traditionally been the lack of knowledge about the surface relaxation parameter, ρ . This has usually been accomplished by anchoring the relaxation time distribution to other measures, such as mercury capillary data or data from image analysis. There are two major disadvantages connected to this procedure. Firstly, the distribution is no longer independent as it relies on information from a second measurement, and secondly the information from the additional experiment may contain different information than the NMR experiment, so equating them can produce significant errors. For example, anchoring the NMR distribution to a distribution from mercury capillary data in practice means equating a pore throat distribution with a pore body distribution.

In recent years several authors has taken advantage of the time-dependency of the apparent diffusion coefficient as measured by pulsed gradient NMR to provide an independent measure for the surface-to-volume ratio of the porous medium. (Fleury 2007; Slijkerman and Hofman 1998; Sorland et al 2007; Uh and Watson 2004) According to Mitra et al (1993), at short time diffusion times the reduction in the apparent diffusion coefficient is proportional to the square root of time, the constant of proportionality being the surface-to-volume ratio of the porous medium.

$$\frac{D(t)}{D_0} = 1 - \frac{4}{9\sqrt{\pi}} \left(\frac{S}{V}\right) \sqrt{D_0 t}$$
⁽²⁾

Under the assumption that the surface relaxivity is uniform it is then possible to solve equations 1, 2 for the surface relaxivity so to transform the measured T_2 distribution in to a true pore size distribution.

In addition to these two methods, x-ray micro computed tomography was also applied to one of the samples. As is for NMR, computed tomographic measurements are nondestructive to the sample, and have a non-percolative nature. Micro computed tomographic data also holds the advantage that there is no limitations for large pores as can cause problems for both NMR and mercury derived pore size distributions. In addition, the segmentation of the tomographic data provides the basis for a whole set of network statistics complimentary to the pore size distribution, including separate pore body and throat distributions, body to throat aspect ratio as well as network connectivity.

RESULTS AND DISCUSSION

The samples selected for this study are taken from an outcrop in the Prebetic subzone of the Betic Range (Spain), and is characterized by a high degree of heterogeneity, containing significant amount of vugs in the millimetre range. Previous studies has shown an average percentage of vugs to be approximately 65% (Vik et al 2007) based on NMR T_2 distributions and segmented computer tomographic data.

The material was first analysed by mercury injection. Three samples where subject to injection, and the resulting capillary pressure curves turned out to be quite similar. The corresponding pore size distributions are shown in figure 1. The three distributions peak at approximately 70 μ m, and show an average pore size of about 25 μ m. The right cut-off is at 200 μ m. A visual inspection of the material show that there are pores well above this size, so pore size distributions from mercury injection clearly has limitations for this type of material. This can also be visualised through comparing the porosity estimates from the mercury injection data with porosity data from water saturation. The three samples show an average porosity of 21.7 per cent after mercury injection, whereas the average value from water saturated samples is 28.9 per cent (Vik et al 2007). The porosity data indicates that the samples for mercury injection are too small to represent the vuggy portion of the pore space, implying that the resulting pore size distributions correspondingly mainly



Figure 1: Pore size distributions from mercury injection (Three data sets)

reflect the matrix pore space. This is in accordance with the findings of Egermann et al (2007) as well as some of the samples by Ausbrooks et al (2004) and Hidajat et al (2004). The likeness of the three distributions also indicates that the matrix is relatively uniform throughout the samples.

To explore the vuggy portion of the pore space, combined diffusion-relaxation NMR was applied to two samples. The procedure used was described by Sorland et al (2007). Due to solubilisation of paramagnetic components from the pore surfaces into the water phase, all samples were flooded with temperate water immediately prior to experiments. The development of the apparent diffusion coefficient with time was relatively similar for the two samples. Linear regression in the short time regime gave average surface-to-volume ratios ranging from 0.47 to 0.58 μ m⁻¹. Combined with the *T*₂ distributions this resulted in values for the surface relaxivity of approximately 0.04 cm/s. The resulting pore size distributions are shown in figure 2.



Figure 2: Volume-to-surface ratio distributions from NMR (Two data sets)

The calculated value for the surface relaxivity is among the highest values reported, though still in the same order of magnitude as values reported by Carr et al (1996) and Howard et al (1993). The high value can be explained by the paramagnetic content in the material under study. In addition, several of the reported values for the surface relaxivity for similar materials are based on equating mercury capillary distributions with the relaxation time

distribution from NMR. As noted previously, this can lead to erroneous interpretation, and consequently a too low value for the surface relaxivity as the pore volume distribution is assigned to the properties of a throat distribution. The distributions for the different samples again show the same internal characteristics, but compared to the pore throat distribution from mercury injection there is now a large peak representing the vuggy pore space. This is again similar to what Egermann et al (2007) saw when their samples was subject to low-field NMR experiments. The left shoulder of the distribution is in accordance with the shape of the distribution from mercury injection, strengthening the hypothesis that the pore throat curve mainly reflects the matrix pore space.

To confirm the results from the normalized NMR distribution, it can be useful to compare the results with the pore size distribution from network statistics obtained from segmented tomographic data. The resolution in the x-ray micro tomography experiments sets a limit for the smallest pores measureable. As seen in the distribution for the volume weighted pore volume equivalent radius of one of the samples from the NMR experiment, shown in figure 3, pores below approximately 60 μ m are not resolved. The pore size distribution from the segmentation resembles that part of the NMR derived distribution which corresponds to large matrix pores and vuggy pore space. This is also confirmed through omparing the porosity data. The porosity is 24 per cent according to the segmentation whereas the value for the same sample after water saturation which is what the NMR experiment measures is approximately 31 per cent. This difference corresponds to the part of the NMR distribution that lies below the resolution of the tomographic data.



Figure 3: Pore volume equivalent radius distribution from μ -CT

CONCLUSIONS

As part of a larger study on vuggy carbonate material different measures of pore size have been explored. For this type of heterogeneous media and correspondingly large pore size range it is evident that there is a significant difference between an invasion based method as mercury capillary pressure curves and methods based on measuring a saturated sample as a whole. Mercury injection is sensitive to the matrix part of the pore space, but may erroneously assign vugs and microfractures to the matrix pore space. NMR derived distributions do to a larger extent describe the whole range of void spaces, as confirmed by μ -CT data. It should be emphasised that mercury intrusion, NMR and μ -CT are all complementary analysis as part of the process of understanding the pore system, each method carrying information not inherent in the others. To get a complete description of the porous system all methods should be used supplementary to each other.

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