CHARACTERIZATION OF VUGGY CARBONATE BY MISCIBLE PROCESSES

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ABSTRACT

This paper addresses connectivity and representative volume for miscible displacement for vuggy carbonate material in which the porosity is divided into matrix and vuggy pore space. The results are based on effluent tracer tests and Nuclear Magnetic Resonance (NMR) measurements of water (H2O) and deuterium oxide (D2O) flooding.

The different intrinsic flow velocities and different flow paths are the main reason for dispersion in porous media. Thus, the dispersion characteristics are assumed to be affected by connectivity and local heterogeneity of the material which also leads to questions about the representative elementary volume for miscible fluid flow properties.

In this study dispersion tests at the macroscopic scale have been performed on heterogeneous vuggy carbonate material. The dispersion characteristics have been measured at different length scales for the vuggy material and quantified by the Coats and Smith capacitance model. The length range of the studied material is from cm to m scale.

Inverse modelling was used to fit the experimental data and to determine the values for the dispersion coefficient, the flowing fraction and the mass exchange constant as described by the capacitance model. The determined values are considered as an average for the tested samples and are subjects for the discussion on representative elementary volume. It is shown that with decreasing sample length the characteristics deviate from mean values for the material. H2O and D2O miscible flow has been performed using NMR measurements under dynamic conditions to determine which pore sizes belong to the flowing and dendritic fractions.

INTRODUCTION

Hydrodynamic dispersion occurs when a miscible fluid displaces another and particles injected into the displacing fluid spreads in time and space. Dispersion is of importance when dealing with enhanced oil recovery methods as miscible and surfactant flooding but also for transport of contaminants in underground water. By a better understanding of dispersion phenomena in porous media the design of the above mentioned processes can be improved.

Gnist *et al.* (1990) concluded that prediction of dispersion properties in carbonate rock is far more difficult than in sandstones. Particularly, heterogeneities for carbonate rocks at the laboratory scale will be different at a larger scale, and therefore the dispersion characteristics will deviate between the different scales. In our work the experimental measurements of the dispersion characteristics have been performed up to m scale where little or no work has been done previously.

Laboratory dispersion tests on different scales are performed by monitoring the effluent concentration of the tracer injected into the sample. The longitudinal dispersion coefficient is determined by fitting the effluent concentration characteristics to the Coats and Smith model. In addition, each curve provides us with two other constants existing in the capacitance model, which are the flowing fraction and the mass exchange. The values determined are average values, and include the contribution of local heterogeneities within the sample. Measurements have been done for different sample lengths and cross sections.

Short introduction to the Coats and Smith capacitance model

The Coats and Smith (1964) capacitance model is described by the following equations:

$$D\frac{\partial^2 C}{\partial x^2} - v\frac{\partial C}{\partial x} = f\frac{\partial C}{\partial t} + (1 - f)\frac{\partial C^*}{\partial t}$$
(1)

$$(1-f)\frac{\partial C^*}{\partial t} = M(C-C^*)$$
⁽²⁾

where u, D and C are the interstitial velocity, the dispersion coefficient and the concentration in the pores occupied by the fluid. M is the mass transfer coefficient between the dendritic regions and the flowing fluid, while f is the dimensionless flowing fraction of accessible fluid. C^* is the concentration in the stagnant fraction, x is distance and t time. Earlier studies have shown that both molecular diffusion and mechanical or convective mixing contribute to the dispersion coefficient. The equation given below expresses this relationship for the dispersion coefficient:

$$D = \frac{D_o}{F\phi} + \alpha v \tag{3}$$

where D_o is the effective molecular diffusion coefficient between the miscible displacing and displaced fluid, F is the formation factor, ϕ is the porosity, α is the dispersivity and v is the interstitial velocity.

Under the assumption that the effect of molecular diffusion is negligible, further simplification as found in the groundwater literature by Bear, (1972) and Domenico and Schwartz, (1990) gives:

$$D \approx \alpha v$$
 (4)

Model limitations

The Coats and Smith's model is a 1D model that does not take into account porosity variations within the sample. However, a dispersion model involving porosity heterogeneities would require numerical solutions, as finite-differences leads to punctilious core description which is still an unsolved problem. In addition there is a lack of proper permeability-porosity correlation. The Coats and Smith's model is used in this study because it has an analytical solution that makes parameter estimation straight forward based on the effluent characteristics.

According to Fourar *et al.* (2005), the average dispersion coefficient found in all standard approaches is no longer applicable because the dispersion coefficient depends on space location. These variations are caused by local porosity and permeability heterogeneities, as is the case with heterogeneous vuggy material. Therefore, it is important to point out that the determined dimensionless parameters in this study are considered as an average for the tested samples. Earlier studies revealed the Coats and Smith model's limitations. The limitations should be considered as this model is applied for parameter estimation in the study of effluent characteristics.

Salter and Mohanty (1982) reported problems fitting the experiments to the model, resulting in either very low f or f's near unity. Non-uniqueness was the problem of the high f solution. They also pointed out that the model holds only one mass transfer, whereas in real porous media there are a series of dendrites appearing with varying sizes and shapes. Moreover, the mass transfer is an effective average parameter that is independent of the concentration gradient between flowing and dendritic fraction.

Laboratory set up

For most of the effluent dispersion tests reported in this study, the tracer concentrations at the outlet were found by measuring the fluid electrical resistivity over a non-conducting tubing. Initially the tested cores were saturated with brine. The injected tracer was brine with additional 1wt% NaCl with higher electrical conductivity compared with the initial fluid in place. The temperature of the effluent is also monitored as it influences the resistivity at a given concentration. An a priori calibration of the setup relates conductivity and temperature to the effluent tracer concentration.

Compared to UV spectrometry the resistivity method is favorable as it prevents adsorption, decreases the uncertainty as it averages the concentration over smaller injected volumes, and increases the amount of data points significantly. In addition, the analysis time is reduced. Figure 1 shows the experimental setup schematically.



Figure 1. The experimental set-up used for dispersion measurements. Tracer is injected from left and the effluent resistivity and temperature is measured at the outlet.

Some of the early effluent experiments were performed using UV spectrometry after injection of brine with 4-fluoro benzoic acid (4-FBA) as the tracer. When comparing the two methods on the material studied here the results are similar, but as mentioned above, the UV spectrometry introduces more uncertainty and less data points compared to the resistivity measurements.

All effluent experiments were started at 100% brine saturation following injection of tracer and run until produced fluids were at the injected fluid concentrations. This occurred usually at 3-3.5PV injected. The viscosity ratio for the fluids used in both methods is assumed to be near unity as the tracer additives have negligible effect on the fluid properties.

NMR profile imaging

Miscible displacement studies have been used to investigate heterogeneity in porous media. Studies by deZabala and Kamath (1995), and Hidajat *et al.* (2004), pointed out the existence of heterogeneities and preferential flow paths in vuggy carbonates from tracer concentration profiles. The preferential flow path was found to affect the oil recovery process, and some of the tests also indicated that a significant portion of the pore volume was not well connected to the main flow paths deZabala and Kamath (1995).

Miscible tracer tests can also be analysed by monitoring the changes in the in situ concentrations using x-ray CT (Peters *et al.* (1996); Fourar *et al.* (2005); Hicks *et al.* (1994)). Using this technique, Hicks *et al.* (1994) tracked the dominant flow paths through a heterogeneous carbonate core and found them to be where the core porosity was highest. Peters *et al.* (1996) and Fourar *et al.* (2005) measured the longitudinal dispersion coefficient by imaging a sample during tracer test using computed tomography (CT). As the traditional effluent tracer test only gives concentration at the outlet the imaging of a tracer can be used to find concentrations in space and time. Both Peters *et al.* (1996) and Fourar *et al.* (2005) concluded that imaging the tracer test allows the contribution of sample heterogeneity to the dispersion mechanism to be determined.

In this work the concentration profiles as a function of space and time for the miscible displacement have been found measuring 1D NMR profiles. The displaced and displacing fluids in experiments are H2O and D2O respectively. All NMR experiments were performed on a 12 MHz NMR instrument at 35°C and ambient pressure. Because

deuterium has different Zeeman energy levels (Friebolin, 2005) than hydrogen it does not contribute to the NMR signal at the proton resonance frequency generated by the RF source. Thus, the NMR signal is generated only by the H2O component during the miscible process and concentrations in space and time may be calculated.

1D profiles resolving the NMR signal spatially along the sample can be acquired using a profile pulse sequence. When subject to a homogenous magnetic field, all hydrogen protons throughout the whole sample will resonant at a similar frequency, giving no spatial information. Instead, applying a magnetic field gradient provides spatial information about the signal from the Fourier transform of the signal. Additional information regarding acquisition of NMR 1D profiles beyond the scope of this paper can be found elsewhere, Callaghan (1991).

When injecting D2O the profiles change, and reflect the concentration of water along the core sample. Compared to CT this method provides faster measurements of the concentration profile at a better resolution. Measurement time for a profile determination is about 60 seconds with a resolution about 0.7mm. Since the miscible displacement experiments are performed under dynamic conditions, measurement time is of importance and a contributor to profile uncertainty. This method is favorable as it prevents adsorption and increases the amount of data points significantly. The effective molecular diffusion coefficient, D_o , for D2O and H2O are similar according to Eisenber *et al.* (1969). Gravity segregation between H2O and D2O was investigated; it was found to be negligible.

RESULTS

Effluent dispersion tests performed on 20 vuggy material samples were quantified by parameter estimation of the Coats and Smith capacitance model. The length range of the studied material is from cm to m scale, including both circular and rectangular cross sections. Permeability for this material is in order of 30-40Darcy. The estimated longitudinal dispersivity values as a function of sample length are presented in figure 2 below.



Figure 2. The estimated dispersivity values as a function of sample length.

As seen from the figure 2 above, the plot shows scattering for the shorter sample lengths, as is expected for heterogeneous material. However, the longitudinal dispersivity values for sample lengths greater than 30 cm tend to follow a linear relationship, earlier reported by Arya *et al.* (1988). The results imply the representative length for miscible displacement to be about 30 cm. It is also evident that the Coats and Smith model captures the expected trends in relation to sample lengths.

NMR profile imaging was performed on a 4.46 cm long, 3.76 cm in diameter and 29.7% porosity, core sample at two flooding rates, 0.006 and 0.003 ml/s corresponding to an average intrinsic velocity, v, of 0.002 and 0.001 cm/s, respectively. The in situ D2O concentration, C, is presented as the fraction of the injected D2O tracer, C_o , giving a dimensionless tracer concentration C/C_o . Profiles of dimensionless tracer concentration as a function of time and distance for both rates are presented in figure 3 below.



Figure 3. Dimensionless tracer concentration as a function of time and distance for v=0.002 cm/s (left) and v=0.001 cm/s (right). The first 9 time steps are plotted.

Figure 3 shows profiles for the first 9 time steps from 0.02 to 0.47 total PV injected (v=0.002 cm/s) and 0.02 to 0.35 total PV injected (v=0.001 cm/s). As seen in figure 3, the profiles for the lower rate are relatively flat with early breakthrough compared to higher rate profiles. This illustrates the profile alternation as the miscible process moves into more diffusion controlled regime with decreasing intrinsic velocity. Figures 4 and 5 below show the in situ concentration curves for six cross sections along the core. The positions are numbered in increasing order from the inlet to the outlet. The core is divided into a total of 65 cross sections giving a resolution of 0.69 mm.



Figure 5. Dimensionless in situ tracer concentrations at different positions along the core. v=0.001 cm/s

Both experiments show that the in situ concentration curves are moving towards an average value for the samples with increasing dimensionless position. The curves tend to become more similar towards position lengths near the end of the sample. The shortest positions from the outlet are strongly influenced by the heterogeneities and preferential flow path connected to the inlet. Similar results have been reported by Fourar *et al.* (2005). For the effluent dispersion test at different scales the samples were flooded until produced fluids were at the injected fluid concentrations. This usually occurred at 3-3.5 PV injected. However, the D2O tracer tests show that the in situ concentrations remain lower than the effluent concentration and that the fluids are totally exchanged after about 8 PV. This is in accordance with the theoretical study of Brigham (1961), where it is shown that the effluent concentration is always higher than the average in situ concentration.

Dynamic NMR T₂ measurements

To investigate the effect of advection on the NMR T_2 relaxation time the 100% water saturated sample was measured under stationary and flooding conditions. Water was used

as the injected fluid. The measurements show little or no effect on the T_2 distribution for the rates used in this study. The results are shown in figure 6 below.



Figure 6. NMR T_2 relaxation time the 100% water saturated sample at stationary (blue) and flooding (pink) conditions.

As seen from figure 6, the curves representing stationary and flooding conditions are approximately identical. It is important to point out that the observed dissimilarities are mostly related to unstable inversion of the NMR relaxation curve into the T_2 distribution.

In the next section dynamic NMR T_2 measurements are carried out under tracer (D2O) miscible flooding to investigate if different types of miscible displacements can be dedicated to matrix and vuggy pore space. This is done under the assumption that the correlation between the pore size distribution and T_2 relaxation is applicable under flooding conditions, as indicated above.

Idealized connected-vugs: Berea cavity core

To investigate the NMR response on a known bimodal pore size distribution, an experiment was performed on a 4.1 cm long and 3.75 cm in diameter water saturated Berea sandstone core with an introduced tube like cavity, figure 7. The cavity is a centered bore hole drilled along the core length, with a volume of 5.1 cm^3 and a calculated mean radius of 0.6 cm. It can be considered as a large pore connected throughout the sample with significantly higher radius and permeability than originally found in the material. This type of pore size distribution is an idealized bimodal distribution with a large permeability contrast, and can be regarded as analog to connected-vugs type of material. The flooding rate was 0.029 ml/s.

Figure 8 shows the normalized cumulative frequency, corresponding to the total area under the T_2 curve, as a function of PV injected tracer. The data points are normalized to 100% H2O saturated sample.



Figure 7. The Berea sandstone with drilled tube like cavity.

Figure 8. The Berea sandstone with drilled tube like cavity.

As seen in figure 8, the normalized cumulative frequency curve has a breakpoint at about 0.5 PV injected. The steep linear decrease in the normalized concentration, from 1 to 0.55, to the left of PV=0.5 is mainly due to convective motion from the flooding through the cavity channel, as the channel represents 37% of the total pore volume. To the right of the breakpoint, the concentration decreases at a slower rate, caused by diffusion between the cavity channel and the matrix pore space.

It is common to divide vuggy carbonates into two main groups; those containing separate and those containing touching vugs. Vugs are characterised by being significantly larger than the typical size of the particles making up the interparticle porosity. Separate vugs are defined as pore space that is connected only through interparticle porosity, whereas touching vugs are defined as pore space that forms an interconnected system independent of the interparticle porosity Lucia (1999). While the addition of separate vugs increases the total porosity, it does not significantly increase permeability. The presence of touching vugs, on the other hand, increases permeability well above what would be expected from the interparticle pore system Lucia (1983).

Dauba *et al.* (1999) used simulations of miscible displacements in 2D heterogeneous cores to evaluate the effect of different core scale heterogeneities on tracer effluent profiles. The cores were divided into two distinct regions, both of which were treated as homogeneous media with separate hydraulic and solute transport properties. The simulations revealed that cores with cross-section heterogeneities behaved like homogeneous cores, even at high permeability contrast. Along-axis heterogeneities, on the other hand, caused early tracer breakthrough followed by significant tailing. The effect of the heterogeneities was more pronounced at high tracer injection rates since the high velocity leads to less mass exchange between the two regions.

Earlier studies for the material examined in this study (Vik *et al.*, 2007) have concluded an average percentage of vugs to be approximately 65% of the total porosity based on NMR T_2 distributions and segmented computer tomographic data. The question about connectivity between the vugs was still unanswered. Pursuing the idea of vuggy connectivity led to dynamic NMR T_2 measurements on the material where a cut-off between vugs and matrix was established.

Our approach for the dynamic NMR T_2 measurements of the 4.46 cm core, considers the core to be made up of two distinct but internally homogeneous regions, that is vuggy and matrix pore space. Earlier studies for this material (Vik *et al.*, 2007) yield similar pore distribution for the matrix pore space. In an earlier study (Hidajat *et al.*, 2004), where six carbonate cores were studied using NMR T_2 distributions, a cut-off value was introduced to separate between vuggy and matrix porosity. Normalized cumulative frequency corresponding to the total, vuggy and matrix area under the T_2 curve as function of PV injected for v=0.002 cm/s (figure 9) and v=0.001 cm/s (figure 10) are shown below. The cumlative frequencies for the total, vug and matrix area under the T_2 curves are normalized to their respective initial values before injection of displacing fluid.









Consistent with the study of Dauba *et al.* (1999), the figures above show that the effect of the heterogeneities increases with increasing tracer injection rates. Higher intrinsic velocity leads to faster concentration change for the vugs, whereas the concentration change for the matrix is less independent of velocity variation. For the flooding at higher intrinsic velocity, the dominant flow paths are mainly attributed to the vuggy pore space. This indicates that a large fraction of the vuggy pore space forms touching vug channels. The difference in matrix and vuggy behaviour is also in accordance with an earlier study that indicated different properties for matrix and vuggy pore space in terms of residual gas saturation after spontaneous imbibition of water

CONCLUSIONS

Effluent dispersion at different length scales were successfully quantified by the Coats and Smith model. Sample lengths greater than 30 cm tend to follow a linear relationship between dispersivity and sample length.

A new method is presented that by means of 1D NMR profiles, determines tracer concentration as a function of time and distance. The D2O tracer tests show that the in situ concentrations remain lower than the effluent concentration.

Also, a new method for determining the transport properties of vuggy carbonates, based on dynamic T_2 measurements during flooding is presented. Partitioning between matrix and vuggy pore space from the T_2 distributions yields different properties for the vuggy and matrix pore space during miscible displacement at different intrinsic velocities.

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