Using Mercury Intrusion Porosimetry to Identifying the Sample Size Effect on Pore Structure Characteristics of the Eagle Ford Shale

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Abstract

Capable of working with different sample sizes, mercury injection capillary pressure (MICP) is a common technique used for characterizing pore structure (porosity, pore volume, and pore-throat size distribution) of reservoir rocks. Related to pore connectivity, a rock sample can exhibit sample size-dependent porosity; a poorly connected rock could have a larger porosity for a smaller-sized sample, and the porosity reaches a stable value at certain sample sizes. In this study, we focused on Eagle Ford Shale and used Atco Chalk, Del Rio Claystone, and Salmon Peak Limestone as comparisons. Our results indicate that when applying multiple sample sizes (1cm³ cube, crushed granular samples with sizes varying from 1.7-2.36 mm, 500-841μm, 177-500 μm, and 75-177 μm), the resultant porosity dramatically increases (accordingly at 2%, 3%, 6%, 9%, and 33%) for Eagle Ford Shale as an example. Comparing the MICP pore-throat distribution plots of cubic and granular samples, pore systems were generated and destroyed during sample crushing. The degree of pore systems’ generation and destruction is directly related to the sample sizes. The results suggested although the porosities of crushed samples may not have an obvious difference, the pore systems are different from non-crushed samples.

Reference Cited

Using Mercury Intrusion Porosimetry to identify the Sample Size Effect on Pore Structure Characteristics of the Eagle Ford Shale

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Abstract

Capable of working with different sample sizes, mercury injection capillary pressure (MICP) is a common technique used for characterizing pore structure (porosity, pore volume, and pore-throat size distribution) of reservoir rocks. Related to pore connectivity, a rock sample can exhibit sample size-dependent porosity; a poorly connected rock could have a larger porosity for a smaller-sized sample, and the porosity reaches a stable value at certain sample sizes. In this study, we focused on Eagle Ford Shale and used Atco Chalk, Del Rio Claystone, and Salmon Peak Limestone as comparisons. Our results indicate that when applying multiple sample sizes (1 cm³ cube, crushed granular samples with sizes varying from 1.7-2.36 mm, 500-841 μm, 177-500 μm, and 75-177 μm), the resultant porosity dramatically increases (accordingly at 2%, 3%, 6%, 9%, and 33%) for Eagle Ford Shale as an example. Comparing the MICP pore-throat distribution plots of cubic and granular samples, pore systems were generated and destroyed during sample crushing. The degree of pore systems’ generation and destruction is directly related to the sample sizes. The results suggested although the porosities of crushed samples may not have an obvious difference, the pore systems are different from non-crushed samples.

Background

(1) Luffel and Gridly first utilized crushed samples to determine the porosity of tight rock by helium pycnometry in 1992.
(2) Several previous studies reported both porosity and permeability are highly influenced by sample sizes.
(3) The Sample size is not the only control, conformance and micro-fracture were reported as the two major influences.
(4) MICP has the capability to detect pore-throat size distribution. By using this technique, any pore space regeneration and destruction will be detected.

Results

(1) Porosity

<table>
<thead>
<tr>
<th>Sample and analysis conditions</th>
<th>1.7-2.36 mm</th>
<th>500-841 μm</th>
<th>177-500 μm</th>
<th>75-177 μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Before conformance</td>
<td>0.2 psi</td>
<td>5 psi</td>
<td>20 psi</td>
<td>50 psi</td>
</tr>
<tr>
<td>After conformance</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Eagle Ford Shale</td>
<td>2.074%</td>
<td>2.035%</td>
<td>3.262%</td>
<td>6.510%</td>
</tr>
<tr>
<td>Del Rio Claystone</td>
<td>&gt; 10%</td>
<td>&gt; 10%</td>
<td>16.040%</td>
<td>15.920%</td>
</tr>
<tr>
<td>Atco Chalk</td>
<td>8.995%</td>
<td>6.473%</td>
<td>8.022%</td>
<td>7.276%</td>
</tr>
<tr>
<td>Salmon Peak Limestone</td>
<td>6.778%</td>
<td>7.628%</td>
<td>7.999%</td>
<td>9.278%</td>
</tr>
</tbody>
</table>

(2) Mercury Intrusion Capillary Pressure

(3) The Sample size is not the only control, conformance and micro-fracture were reported as the two major influences.

Discussion

(1) We used quartz sand as non-porous material to investigate the MICP conformance effect. The results show conformance effect on quartz only affect the part which pore size is larger than 10 μm.
(2) Acid-washed quartz was tested in their natural manner, no artificial impaction, all the pore systems detected are estimated as cleavages and natural fractures.
(3) Eagle Ford Shale shows pore volume increasing in the range of 10 μm to 100 nm with the sample size decreasing. But nano-pores are keeping its original manner.
(4) Each of Del Rio Claystone, Atco chalk, Salmon peak limestone show one huge peak between 1 μm to 100 nm. This peak is the major pore system of each rock. With the sample size decreasing, the main peaks were gradually been destroyed. Pore space on both flanks of the peak increasing with sample size decreasing.
(5) Opening of isolated pores are first-order suspect of porosity increasing in shale. Artificial micro-fractures is another suspect.
(6) Density is a very good reference parameter to verify the precision of the MICP test. The grain density of quartz is 2.684 g/cm³, the bulk density of shale is between 2.06 to 2.67 g/cm³, and the bulk density of carbonate is around 2.83. When the density generated from analysis has a large offset, that’s means that analysis is extremely high attention needed.

Further work

Ion-milling SEM will be conducted on both cubic samples and granular samples to detect the artificial micro-fractures.

Acknowledgement

We thank AAPG R. E. McAdams Memorial Grant for the financial support.

Method

1) Sample preparation
   Eagle Ford Shale, Atco chalk, Del rio claystone, and Salmon peak limestone were prepared as 1cm³ cube, crushed granular samples with sizes varying from 1.7-2.36 mm, 500-841 μm, 177-500 μm, and 75-177 μm. Acid-washed Quartz grains were used as blank test to correct conformance.

2) MICP
   (1) Conducted by Micrometrics Auto pore 9500
   (2) Pressure conditions were set from 0.2 psi (Pore throat Diameter 900 μm) and 5 psi (37 to μm) 60,000 psi (2.8 nm).
   (3) Quartz sand was tested in both condition, other four samples were tested from 5 psi to 60,000 psi.

3) Ion-milling SEM
   Ion-milling SEM will be conducted on both cubic samples and granular samples to detect the artificial micro-fractures.

Reference