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STRESS SENSITIVITY OF MERCURY INJECTION MEASUREMENTS

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ABSTRACT

Many petrophysical properties (e.g. permeability, electrical resistivity etc.) of tight rocks are very stress sensitive. However, most mercury injection measurements are made using an instrument that does not apply a confining pressure to the samples. Here we further explore the implications of the use and analysis of data from mercury injection porosimetry or mercury capillary pressure measurements (MICP). Two particular aspects will be discussed. First, the effective stress acting on samples analysed using standard MICP instruments (i.e. Micromeritics Autopore system) is described. Second, results are presented from a new mercury injection porosimeter that is capable of injecting mercury at up to 60,000 psi into 1.5 or 1 in core plugs while keeping a constant net stress up to 15,000 psi. This new instrument allows monitoring of the electrical

conductivity across the core during the test so that an accurate threshold pressure can be determined.

Although no external confining pressure is applied (unconfined) when using the standard MICP instrument, this doesn't mean that the measurements can be considered as unstressed. Instead, the sample is under isostatic compression by the mercury until it enters the pore space of the sample. As an approximation, the stress that the mercury places on the sample is equal to its threshold pressure. Thus, the permeability calculated from standard MICP data is equivalent to that measured at its threshold pressure. Not all the samples have the same stress dependency thus comparing measured permeabilities at a single stress with values calculated from standard MICP data, corresponding at different threshold pressures, can lead to erroneous correlations. Therefore, the estimation of permeabilities from standard MICP data can be flawed and uncertain unless the stress effect is included.

Results obtained from the new mercury injection system, porosimeter under net stress, are radically different from those obtained from standard MICP instruments such as the Autopore IV. In particular, the measurements at reservoir conditions produce threshold pressures that are three times higher and pore throat sizes that are 1/3rd of those measured by the standard MICP instrument. The results clearly indicate that calculating capillary height functions, sealing capacity, etc. from the standard instrument can lead to large errors that can have significant impact on subsurface characterization.

INTRODUCTION

Mercury injection analysis has been extensively used to estimate the capillary pressure of rocks for the petroleum industry. Initially, measurements were made in an instrument in which core plugs were placed in a core holder with a confining pressure of up to 10,000 psi and mercury was injected manually into the sample at pressures of up to 2,000 psi. It is possible to make electrical measurements during this test so that the pressure at which mercury spans across the length of the sample, often referred to as the *threshold pressure*[1], can be identified. More recently, the trend within industry is to use automated porosimeters that can inject mercury at up to 60,000 psi; this will be referred to as unconfined mercury injection capillary pressure (MICP). MICP is usually conducted on small samples (~1-10 cm^3). Two key criticisms of MICP are that the samples are not placed under a confining stress and that electrical measurements cannot be made to identify a threshold pressure. The latter is important because it is the threshold pressure that is used to calculate petroleum column heights that can be sealed by faults and caprocks (Watts, 1987). Conducting mercury injection experiments without a confining pressure is a particular worry for tight samples whose petrophysical properties are known to be highly stress sensitive. Attempts have been made to pre-

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[1] For a homogeneous rock with a unimodal and narrow pore size distribution the terms entry pressure, threshold pressure and breakthrough pressure are interchangeable. In this paper they will be used with this concept in mind, however, for many natural samples they are different.

stress samples prior to the mercury injection test but only a few results have been published and confining pressures are generally limited to a fixed hydrostatic stress of around 5,000 psi although some measurements at stresses of 16,000 psi have been reported (Mitchell et al., 2003). It has also recently been argued that MICP tests should not be regarded as unstressed measurements because mercury will place an isostatic pressure on the samples, inducing a pore volume compression, until it enters their pore space (Mitchell et al., 2003; Brown, 2015). Indeed, Brown (2015) presented a methodology to take into account the effect that the isostatic pressure has on MICP results when being used to calculate permeability.

The following paper aims to increase understanding of the impact of stress on mercury injection measurements by presenting and analysing results from mercury injection experiments conducted using both an industry-standard instrument and a newly developed mercury injection porosimeter that allows standard core plugs to be confined at very high net stress (up to 15,000 psi) and mercury intruded at pressures of up to 60,000 psi. The new instrument, here referred to as Porosimeter Under Confining Stress (PUCS), which also allows electrical measurements to be made so that the threshold pressure can be identified. The paper begins by describing the samples and methods used including the methodology and analysis process of the new PUCS instrument. The paper compares results from both instruments and discusses use of these results to estimate permeability. Finally, the implications of the results are discussed in relationship to the common uses of mercury injection data such as sealing capacity and saturation height functions.

METHODS

Samples and General Methodology

A range of tight samples were analysed during this study including:-

- Approximately 250 tight gas sandstone samples were analysed; small samples of each were tested using the standard MICP instrument and 18 core plugs tested with PUCS. The samples were mainly from Jurassic, Triassic, Permian and Carboniferous from onshore and offshore Europe but some samples from Australia, Oman and Ukraine were also tested.
- Seven fault rock samples were tested with both methods; four are from faults outcropping in the UK and Miri, Malaysia, three were from core taken from a Triassic reservoir in the Central Graben of the North Sea, UK.
- Three caprocks from petroleum reservoirs of undisclosed locations were tested with both methods.

All samples were supplied as 1.5 in core plugs with off-cuts. Cubes of around 1.5 x 1.5 x 1.5 cm were cut from the offcuts for unstressed Hg-injection analysis. The 1.5 in core plugs were trimmed so that their ends were parallel. The samples were then thoroughly cleaned in a Soxhlet extractor using a 50:50 mixture methanol-toluene or dichloromethane. The samples were then dried in an oven at 65°C until constant weight was obtained. A thorough core analysis program was conducted on each core plug including: (i) X-ray CT using a medical CT scanner; (ii) helium porosimetry only at ambient stress; and (iii) gas permeability vs stress using a pulse decay permeameter during a loading cycle at net stresses of 500 to 7000 psi. The Klinkenberg corrected value was determined by measuring apparent permeability, k_{ap}, at four gas pressures, P_p, and extrapolating plots of k_{ap} vs. $1/P_p$ to $1/P_\mathrm{p} = 0$. The microstructure of all samples were examined using optical and scanning electron microscopy to identify the presence of fractures as well as the key microstructural controls on flow properties (e.g. clay distribution etc.).

Unconfined mercury porosimetry analysis was conducted on all samples using the methodology described below. Mercury injected under stress was conducted on 28 samples (tight gas sandstones, fault rocks and caprocks) using the methodology described later in this section. The unconfined and under net stress porosimetry are both performed in a temperature controlled laboratory at 21 ^{o}C. The preparation of each sample for mercury injection under stress takes approximately two days due to the larger sample size.

Unconfined Mercury Injection

The unconfined mercury injection (MICP) was performed using a Micromeritics Autopore IV 9520 system. This model has four low pressure ports and two high pressure chambers. Clean and dry samples are loaded into a penetrometer and evacuated. The penetrometer is automatically backfilled with mercury. The pressure is then increased to 25 psi (0.17 MPa) in the low pressure port and up to 60000 psi (413MPa) in the high pressure chamber following pre-selected pressures. The change from one selected pressure to the next can be at fixed times or when injection rate becomes less than a user defined value (0.001 µl/g/s was used in this work). The Autopore software does an automatic blank cell correction and data reduction. For more details see the Micromeritics documentation. If necessary a manual volume conformance and bulk rock compressibility corrections (Shafer and Neasham, 2000; Comisky et al., 2011) can be applied during data interpretation.

Mercury Injection Under Stress

A new equipment has been designed to perform mercury Porosimetry Under Confining Stress (PUCS) on competent porous and permeable rocks. A net stress equivalent to reservoir conditions (generally 3000 to 7,000 psia), which is the difference between confining stress and pore pressure, is applied and kept constant on the rock sample during mercury injection. The range of net stress applicable is between 1000 and 15000 psi (6.9 -103 MPa) and the maximum mercury pressure is 60,000 psia (413MPa). The resolution per unit volume of sample of the new equipment is comparable to the Autopore. All aspects of control as well as data collection and display are automatic and processed by computer software. The bespoke software for this system was developed in

Labview by InfLogik. The post processing and data reduction of the data collected during the experiment is dealt with separately in Excel.

Overview Of The Analysis Process

A clean and dry sample is prepared, sleeved and loaded between two metal end platens before beginning the analysis. The first phase is the evacuation of the rock sample and filling the upstream sample assembly volume with mercury. The second phase consists of placing the sample assembly in the pressure vessel and a confining pressure equal to the reservoir net stress is applied for approximately 12 hours (overnight). A sample information file that describes the sample and gives the analysis conditions and other parameters is loaded into the software. Separate files are also loaded to define the pore pressure table, which lists the pressure points at which data are collected during the loading and unloading cycle. The system is initialised and the software automatically controls both mercury and confining pressure whilst recording both volumes. Mercury is injected from one end of the sample and once each mercury pressure point is reached the flow rate is monitored until it becomes less than a pre-set value (typically $0.001\text{mm}^3/\text{cm}^2/\text{s}$) the pressure and volumes are recorded. The top and bottom of the sample assembly are electrically isolated and before mercury injection the core sample is a non-conductor so there is a very large resistivity across the sample. As soon as the mercury spans the length of the sample the conductivity is significantly increased, which is used to accurately determine the threshold pressure.

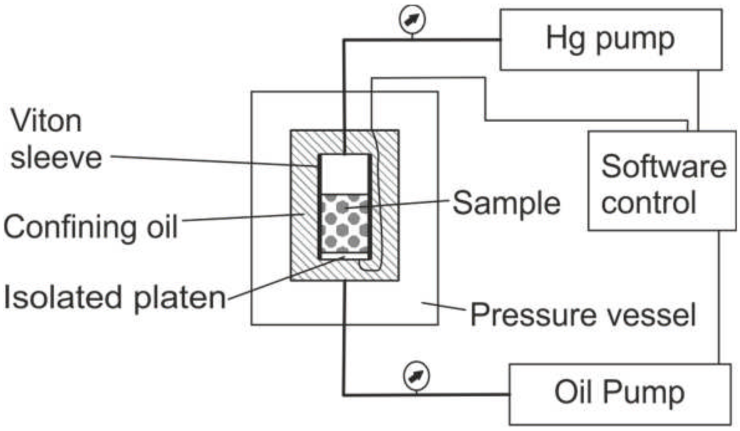


Figure 1. Schematic diagram of the new porosimeter under confining stress.

The data collected during the test is manually processed to obtain capillary pressure as a function of saturation and pore throat size distribution. The porosity under stress is calculated using volume of mercury injected at maximum pressure (pore volume), the sample weight and grain density (grain volume). The pore volume at net stress is calculated by determining the volume injected corrected for system and mercury compressibility effects. The pore diameter at each pressure is calculated using Washburn (1921) equation.

RESULTS

Comparison of Methodologies

To validate the methodology of the new instrument a standard ceramic sample from Soilmoisture Equipment Corp. (15 Bar) was tested in both the Autopore and PUCS. The properties of ceramic disks are likely to be far less stress dependent than core material as they haven't experienced the dramatic changes in stress that core samples experience during extraction. A disk of 38 mm diameter and 5 mm thickness (~5.5 cm^3) was used, to minimize the stress effect in the new system and a sample of 2 x 1.5 x 0.5 (~1.5 cm^3) was used for MICP. A very good agreement between results of both systems was obtained and shown in Figure 2. A threshold pressure of 4000 psig at a saturation of 24.5 % was determined using the resistivity measurement in the new instrument.

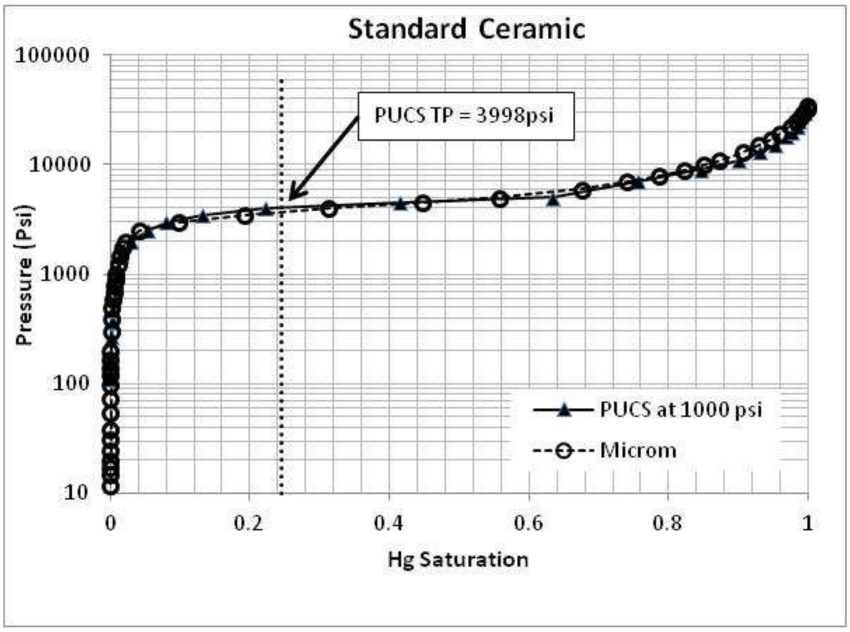


Figure 2. Plot of pressure vs. Saturation for comparing MICP and PUCS. Unconfined MICP (open circles) and the new PUCS stressed Hg porosimeter (filled triangles) . The vertical dotted line marks the saturation at PUCS threshold pressure (TP) as measured using the electrical conductivity measurement.

Permeability Estimates From Unconfined MICP Data of Tight Gas Sandstones

Purcell (1949) was the first to estimate permeability from MICP data by assuming that flow could be calculated applying Poiseuille theory to a bundle of capillaries whose diameter was estimated using the Washburn (1921) equation. The equation of Purcell (1949) contained a term referred to as the lithology factor to account for tortuosity but was obtained by calibration against samples with known permeability. Katz and Thompson (1986, 1987) used percolation theory to derive a method for calculating permeability from MICP data without the need for calibration. Comisky et al. (2007) presented a comparative study of the accuracy of various methods to calculate permeability of tight gas sandstones from MICP data and found that the Purcell (1949) and Katz and Thompson (1986, 1987) performed the best. The current study tested many of the methods including Swanson (1981) and found that all predicted permeability to a similar level, only the results from the method of Swanson are presented here.

A conformance correction was applied to all MICP results by removing any data before true mercury intrusion occurs. Permeability was calculated from the injected mercury data using the equation of Swanson (1981);

$$K_{gas} = A\left(\frac{S_{Hg}}{P_c}\right)_{apex}^{B} \tag{1}$$

where K_{gas} is the gas permeability (mD), S_{Hg} is the mercury saturation (%) and P_c is the capillary pressure (psi) corresponding to the apex of a hyperbolic log-log MICP injection plot. The constants A and B are fitting parameters, which Swanson suggested were 339 and 1.691 respectively. As suggested by Pittman (1992), the apex was obtained by plotting Hg saturation against (Hg saturation/capillary pressure). In the current study, the Excel solver was used to optimize the constants A and B in order to provide the best fit with the Klinkenberg corrected permeability measured at a net confining pressure of 5000 psi. The optimal value of A and B that produced a correlation close to 1:1, between the estimated and measured gas permeability (Figure 3a), are 26 and 1.63 respectively. It should, however, be noted that the method appears to systematically underestimate the permeability of many of the low permeability samples (i.e. <0.0001 mD).

It is often argued that MICP analysis is an unstressed measurement because no confining pressure is applied to the sample during the analysis. However, this is not strictly true as the mercury actually applies an isostatic pressure before it enters the pore space, which becomes important for samples with a high entry pressure. Brown (2015) argued that many of the mercury injection based permeability predictors are broadly based on the assessment of the pore throat diameter of the key pore systems that control flow. So as a first approximation, the permeability values obtained can be regarded as being equivalent to a stress at which the mercury spans across the pore system. The threshold pressure usually increases as the pore size of the network decreases, so a permeability estimated from MICP data of tight rocks should be compared to permeability measured at high confining stress. On the other hand, for high permeability samples it should be compared to permeability measured at lower confining stress. The isostatic pressure effect can easily explain why the permeability estimated using MICP and the method of Swanson is generally lower than measured values for the low permeability samples.

To test the concept presented by Brown (2015) the permeability of tight gas sandstones at a stress equivalent to the mercury threshold pressure was obtained from the stress vs gas permeability data. The Excel solver function was then used to estimate the optimal values of A and B to produce the best correlation between the Klinkenberg corrected gas permeability at stress and the value estimated using the Swanson method. The values of 500 and 1.8 for the constants A and B were found to produce the best 1:1 correlation between measured and estimated values (Figure 3b). The correlation coefficient is similar to that produced when plotted against gas permeability measured at 5000 psi net confining pressure but there is no underestimation of permeability for the tight rocks.

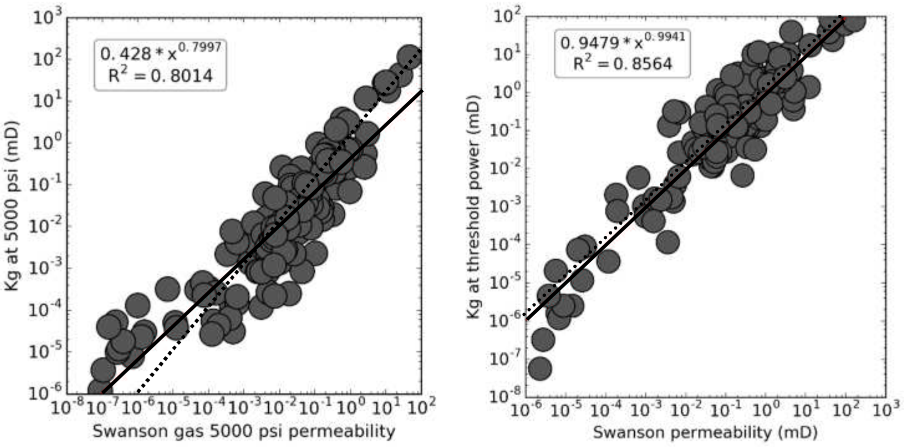
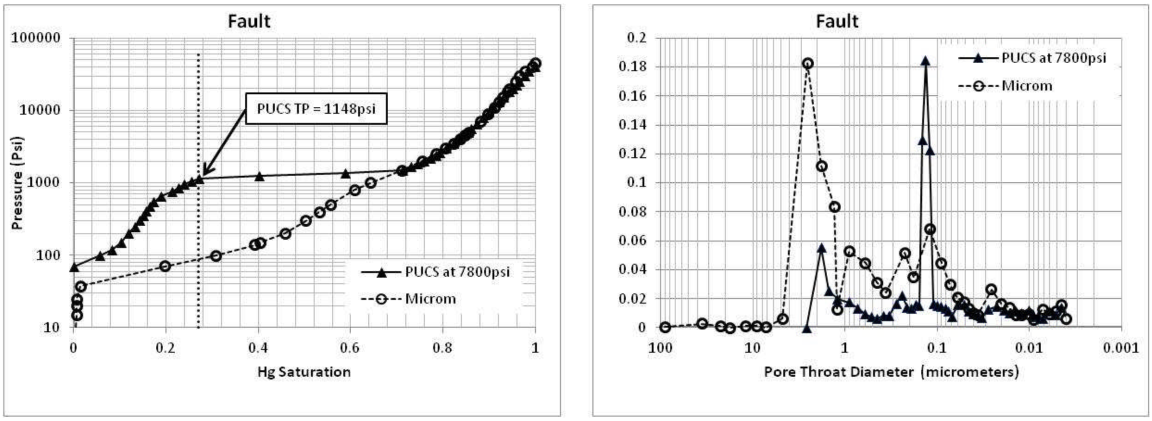


Figure 3. Plot of a) permeability estimated using MICP and the Swanson method (Eqn.1) against Klinkenberg gas permeability measured at 5000 psi confining pressure and b) permeability estimated using the Swanson method

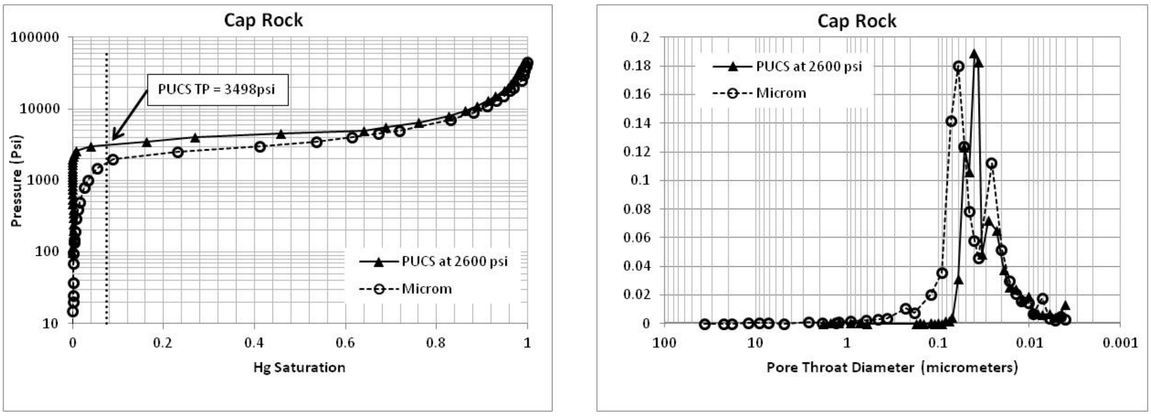
against the gas permeability measured at the mercury-air threshold pressure of each sample. The dashed lines represent the 1:1 relationship whereas the solid lines are the power-law regressions

Stressed Mercury Injection Results of Tight Gas Sandstones

The PUCS results at reservoir net stress are radically different from those obtained using the unconfined MICP (e.g., Figure 4). For example, the threshold pressures are an average of 3 times larger for the stressed PUCS compared to the unconfined MICP (Figure 5a) and the peak pore throat diameters are on average a third of the values for the unconfined MICP (Figure 5b). An unequivocal test has not yet been identified to be absolutely certain that these differences are totally due to stress-related variations in pore structure as opposed to differences in experimental details (e.g. sample size, pressure steps etc.). However, the results are entirely consistent with the stress-dependence of permeability of the samples (e.g. pore size is reduced by a factor of 3 and permeability is reduced by an order of magnitude). In addition, as discussed above, the results obtained from the ceramic disk are very similar for the PUCS and MICP instruments.


Fault
Pressure (Psi)
100000
10000
1000
100
10
PUCS TP = 1148psi
PUCS at 7800psi
Microm
0
0.2
0.4
0.6
0.8
1
Hg Saturation
Fault
0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
PUCS at 7800psi
Microm
100
10
1
0.1
0.01
0.001
Pore Throat Diameter (micrometers)

a)


Cap Rock
Pressure (Psi)
100000
10000
1000
100
10
PUCS TP = 3498psi
PUCS at 2600 psi
Microm
0
0.2
0.4
0.6
0.8
1
Hg Saturation
Cap Rock
0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
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PUCS at 2600 psi
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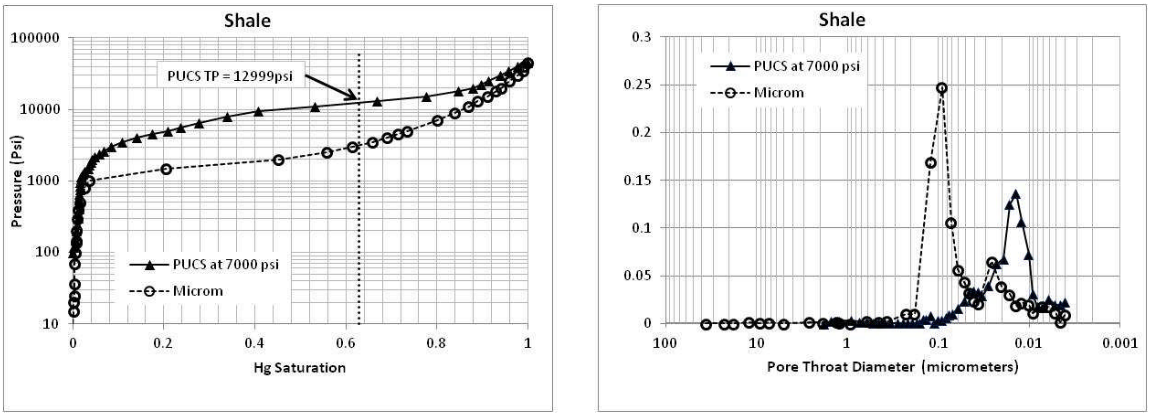
Pore Throat Diameter (micrometers)

b)


Shale
Pressure (Psi)
100000
10000
1000
100
10
PUCS TP = 12999psi
PUCS at 7000 psi
Microm
0
0.2
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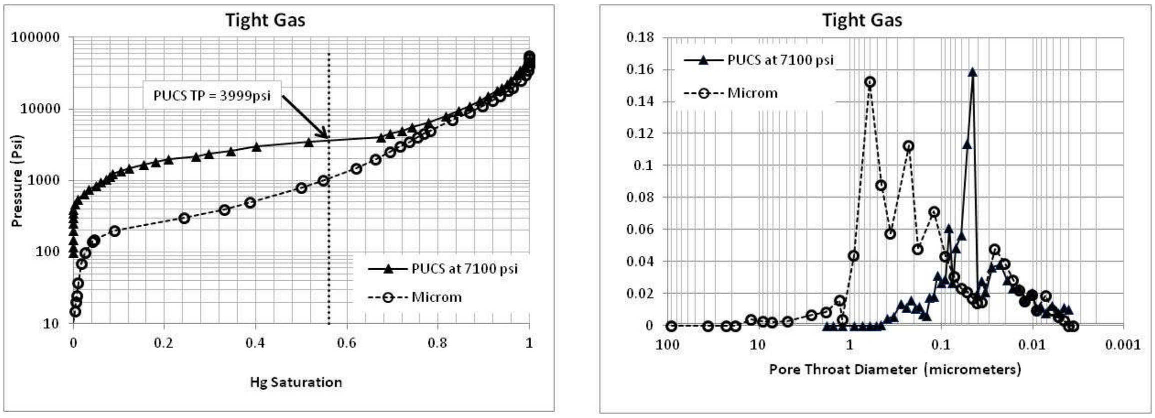
0.6
0.8
1
Hg Saturation
Shale
0.3
0.25
0.2
0.15
0.1
0.05
0
PUCS at 7000 psi
Microm
100
10
1
0.1
0.01
0.001
Pore Throat Diameter (micrometers)

c)



d)

Figure 4. Typical mercury injection results for a) Fault, b) Cap rock, c) Shale and d) Tight Gas samples. MICP (open circles), PUCS (filled triangles) . The vertical dotted lines mark the saturation at PUCS threshold pressure (TP) as measured using the electrical conductivity measurement.

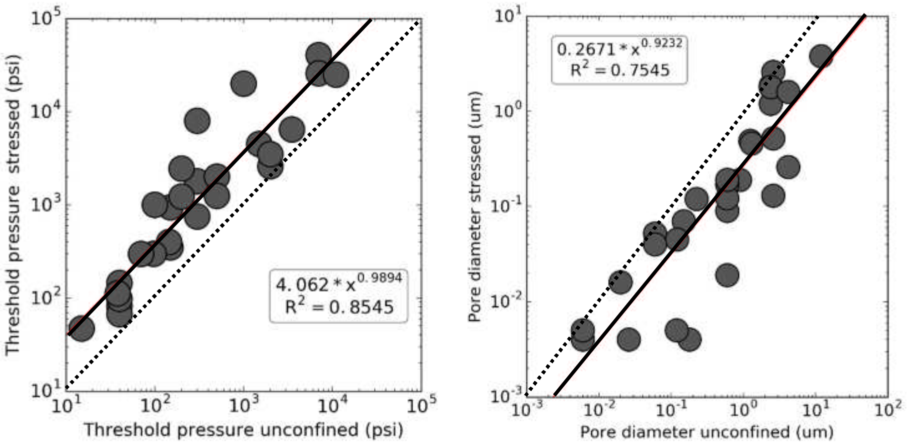


Figure 5 Plot of a) threshold pressure of tight gas sandstones measured PUCS vs MICP, and b) peak pore throat diameter; the dashed lines represent the 1:1 relationship whereas the solid lines are the power-law regressions.

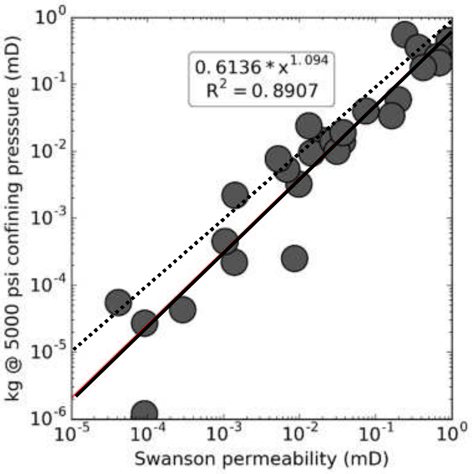


Figure 6 Plot of permeability calculated using PUCS data and Swanson’s method vs the Klinkenberg corrected gas permeability measured at 5000 psi net confining pressure. The dashed represents the 1:1 relationship whereas the solid is the power-law regressions

The Excel solver function was then used to estimate the optimal values of A and B to produce the best correlation between the Klinkenberg gas permeability at and the value estimated using the Swanson method. The values of 560 and 2.05 for the constants A and B were found to produce the best 1:1 correlation between measured and estimated values (Figure 6). The correlation coefficient is slightly better to that produced when plotted against gas permeability measured at net stress of 5000 psi but there is no systematic underestimation of permeability for samples with K_{gas} of <0.001 mD.

Threshold Pressures of Tight Gas Sandstones, Fault Rocks and Top Seals

The threshold pressure represents the capillary pressure at which a non-wetting phase will start to flow and is useful for identifying the sealing capacity of seals and faults as well as the height above the free water level that the critical gas saturation is reached. Figure 7 shows a plot of threshold pressure measured by PUCS at reservoir conditions against that estimated from unstressed MICP data. It shows that on average the threshold pressure measured by PUCS is over four times that estimated from MICP data. However, the threshold pressure at reservoir conditions can be up to an order of magnitude higher that the estimated by MICP.

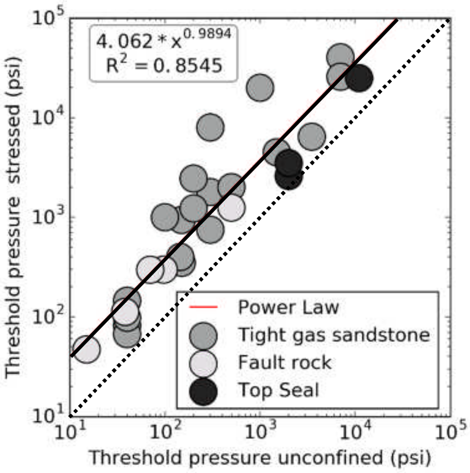


Figure 7. Plot of threshold pressure measured using PUCS against that estimated from MICP data. The dashed represents the 1:1 relationship whereas the solid is the power-law regression.

DISCUSSION

Mercury injection porosimetry is one of the most widely used experimental methods to estimate the capillary pressure characteristics of reservoirs and seals; it is also often used to estimate permeability. Like many other petrophysical properties, MICP results are stress sensitive. The stress sensitivity of many properties (e.g. electrical resistivity, permeability, capillary pressure, etc.) tends to be proportional to pore size. This relationship is not so straight forward for MICP results. In particular, despite often being perceived as an unstressed measurement, the traditional high pressure unconfined measurement, will place an isostatic pressure on the sample inducing a pore volume compression before entering the pore space. The mercury will place a stress of at least the entry pressure onto the sample. This means that mercury injection data conducted on low permeability samples, with a high entry pressure, will have effectively been measured at a higher stress than those of high permeability samples.

An example of the determination of isostatic pore volume compression and entry pressure for MICP has been presented for an Eagle Ford Shale by Comisky et al. (2011). They used a bulk compressibility model to separate the conformance correction, pressure range 10 to 30 psi, isostatic pore volume compression (up to 4000 psi) and intrusion volume (4000 to 60000 psi). Thus, the entry pressure for their shale is 4000 psi but the estimated threshold pressure is 18000 psi. However, based on the depth of their logs the reservoir is approximately at a net stress of 5000 psi and as a consequence none of the properties estimated from MICP (mainly porosity, permeability and capillary pressure) are representative of the reservoir due to an excessive isostatic compression.

There is currently not an agreed method to stress correct capillary pressures obtained from MICP experiments. However, the assumption that the permeability estimated from MICP data using methods such as Swanson (1981) is equivalent to the permeability measured at the mercury injection threshold pressure appears to improve interpretations for the samples used in this paper.

Threshold pressures obtained under constant net stress are on average four times higher than those estimated from traditional MICP measurements. These results are highly significant in that standard MICP data may have been underestimating the sealing capacity of faults and top seals by at least a factor of four. The results also indicate that using traditional MICP data to estimate saturation height functions in tight gas reservoirs could result in a significant overestimation of mobile gas.

CONCLUSIONS

The results from mercury injection analysis are very sensitive to the net stress applied to the sample. Traditional high pressure mercury injection analysis is often thought of as being an unstressed measurement but this is not the case as the mercury provides an isostatic compression to the sample before it enters its pore space. Therefore, if it is assumed that the permeability estimated from the traditional MICP corresponds to the permeability measured at a net stress, equivalent to the sample threshold pressure, a better correlation over a wider range is obtained.

A new mercury porosimeter that performs the analysis under constant net stress has been built and tested. The results indicate that for tight gas sandstones the threshold pressure under reservoir conditions are three times higher than those estimated using the traditional high pressure mercury porosimeter which operates under unconfined conditions. While the average for all the rocks tested is over four times higher. These results are significant when calculating both sealing capacities and saturation height functions. We have not yet attempted to use the results to assess whether it is possible to stress correct unconfined MICP data.

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NOMENCLATURE

A = Swanson fitting parameter

B = Swanson fitting parameter

k_{ap} =*apparent permeability*

K_{gas} = *gas permeability*

MICP = mercury injection capillary pressure

P_c = *capillary pressure*

PUCS = porosimeter under confining stress

S_{Hg} = *mercury saturation (%)*

TP = threshold pressure

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ABOUT THE AUTHORS

Philip Guise graduated in Applied Physics from Brunel University. He then spent nearly 30 years at the University of Leeds in the geochronology and isotope geology laboratories. His expertise in instrumentation and analytical techniques was particularly focussed on Argon dating methods. In 2004 he joined Rock Deformation Research Ltd to assist in setting up a laboratory at the University of Leeds for the study of fault rock petrophysics. Subsequently this has developed into the Wolfson Multiphase Flow

Laboratory. After taking semi-retirement in 2014 he continues his involvement, particularly in the development of the PUCS mercury system.

Carlos A. Grattoni is currently Principal Experimental Officer at the Wolfson Multiphase Flow Laboratory, University of Leeds. He has more than 30 years' experience in laboratory core analysis and the determination of a wide range of petrophysical properties, with a strong research record. After graduating as Chemical Engineer and Reservoir Engineer in Argentina he joined the Argentinian National Research Council (CONICET) becoming a Scientific Investigator. He moved to the UK to study at Imperial College London, where he was awarded a PhD in 1994. He spent 12 years in the Petroleum Engineering Group, Imperial College, working in a wide range of research projects sponsored by the oil industry, EPSRC, EU, and managed the Petroleum Engineering Research Laboratory for five years. In 2006 joined Rock Deformation Research Ltd to develop a laboratory for low permeability rocks while conducting core analysis on fault rocks. In 2012 he joined the School of Earth and Environment as manager of Wolfson Multiphase Flow Laboratory, which is a worldwide state-of-the-art facility on the measurement of flow properties of low permeability rocks.

Sam Allshorn joined the Wolfson Multiphase Lab in 2013 having completed a PhD in Leeds in 2009. Samuel has helped in the final development of the PUCS equipment and the complex methodology involved. Samuel provides support to numerous students in many areas of the Wolfson labs and carries out large amounts of research in the broad range of projects undertaken in the labs

Fisher studied geology at Sheffield University after which he gained a PhD in geochemistry from the University of Leeds. Fisher then spent 15 years conducting research and consultancy on the impact of faults on fluid flow in petroleum reservoirs. In 2008, he took up the Chair in Petroleum Geoengineering at the University of Leeds where he has led research projects that integrate workflows and software in the geological, geophysical, geomechanical and petroleum engineering disciplines. His current research interests include: unconventional hydrocarbons; measurement, visualization and data-mining of petrophysical properties; faulting and fluid flow; coupled fluid flow-geomechanical modelling; multiphase flow in low permeability porous media.

Adam Schiffer received his Ph.D. in Informatics and Electrical Engineering from the Budapest University of Technology and Economics. He has nearly 20 years experience in controls, automation and signal processing. His work experience also includes automation of high pressure pumps, porosity and permeability measurement, acoustic emission testing.