# PETROPHYSICAL AND MINERALOGICAL EVALUATION OF SHALE GAS RESERVOIRS (A COOPER BASIN CASE STUDY)


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# Abstract

Unconventional shale gas reservoirs are over-mature potential source rocks and possess commercial quantities of hydrocarbons in a mechanism which is different from conventional gas reservoirs. These organically rich shale rocks also known as continuous hydrocarbon reservoirs represent a voluminous, long-term, global source of thermo-genic methane and other hydrocarbon gases and could be referred to as shale gas. Roseneath and Murteree shale formations in Cooper Basin have been identified as potential shale gas reservoirs in South Australia. Core samples from these carbonaceous shales were selected for this study.

Petrophysical and mineralogical characterization of shale gas reservoirs is still a challenge due to ultra-fine grained micro-fabric, micro level heterogeneity and anisotropic characteristics of these sedimentary rocks. Unlike conventional gas reservoirs, shale gas reservoirs have very low effective porosity and micro to nano-scale permeability. Conventional standards applications to characterize unconventional gas reservoirs give contradictory results about the mineralogy and rock characteristics, effective and total porosity as well as estimated brine saturation. These uncertainties in the results raised the question, which tools/standards are suitable for petrophysical and mineralogical evaluation of shale gas reservoir.

This research outcome tried to answer the above questions and tells how mineralogy and rock characterization, total porosity, reasons of very low effective porosity and saturation which play a key role in selection and development of a shale gas play, can be evaluated and estimated using both conventional and unconventional techniques

The Roseneath and Murteree shale formations in Cooper Basin have been identified as potential shale gas reservoirs in South Australia. Two core samples from these carbonaceous shales intercepted in Della4 and Moomba46 wells were selected for this project. Core samples were used for number of reservoir characterization phases namely, a) minerals quantification and rock classification, b) visual identification and density of pores location in grains and matrix, c) their types, size classification and interconnectivity, d) evaluation of effective and total interstitial/intergranular and intragranular porosity. While log data from Della# 4 was applied in various resistivity models for water saturation estimation in Murteree shale.

QEMSCAN (Quantitative Evaluation of Minerals using Scanning Electron Microscopy) aided by X-Ray Diffraction techniques was used for mineral and rock characterization. Based on QEMSCAN analysis, we found that Murteree shale has, 42.78% quartz, 6.75% siderite, 28.96% illite, 14.09% koalinite, 1.91% Total Organic Content and 0.04% pyrite, while rutile, sphalerite and some other silicates minerals were identified as accessory minerals. Computerized Tomography (CT), Focused Ion Beam Milling and Scanning Electron Microscopy (FIB/SEM) were employed for identification of pores, network of fractures and their interconnectivity in scanned samples. FIB/SEM combined with CT scanning significantly enhanced shale rock characterization and reservoir petrophysical quality assessment. Helium porosimetry and mercury injection capillary pressure (MICP) techniques were applied for effective porosity and liquid pyknometry was used to estimate the total pore volume. While water saturation was estimated using wireline logs data from Della#4 well completion report and later correlation of results with data found in well completion reports of recent wells drilled through Roseneath and Murteree shale interval.

QEMSCAN results when correlated with XR-diffraction quantitative analyses are found in good agreement with each other for Murteree shale samples. Total free porosity found to be 2 percent is close to total porosity calculated using Wyllie's formula with Hilchie's correction factor. Stieber formula was applied to estimate clay volume (52.3%) from Gamma Ray logs data and this figure

is close to estimated clays volume in XR-diffraction and QEMSCAN techniques 50.59% and 47.30% respectively. QEMSCAN coloured maps revealed the location of lamination, quartz and clay rich zones, high and low porosity zones as well as high and low sorption areas. FIB/SEM scanned images showed that free porosity is largely associated with clay and siderite rich zones. Pores are intergranular and linear, isolated by clays microfabric lamination, and elongated wedge shaped. All pores types and morphology are described.

Since individual pores and porosity evaluation found in QEMSCAN and FIB/SEM technique are not possible to be imaged using CT-scanning, only a network of micro-fracture system in Murteree shale sample was identified in CT-scanned samples. Therefore, no porosity evaluation is possible using CT-scanning. To check the interconnectivity of the pores and fractures network, helium porosimetry was used to measure effective porosity revealing very low effective porosity less than 2.0 % on all samples. Also MICP techniques revealed that samples were mainly comprised of meso-pore throat sized pores, with pore throat diameters range between 2–50 nanometres and an effective porosity of less than 2%. Pores aperture size was documented by 2D imaged pores size and their interconnectivity in FIB/SEM results. The MICP based pores throat classification confirms ultra-low permeability in these reservoirs. The contradiction between imaged pore sizes and the effective porosity led to estimate the total porosity using liquid pyknometry.

Liquid pyknometry method was applied on crushed/powder samples to measure total porosity. It estimated an average absolute porosity of 30.5% for Murteree shale and 39% for Roseneath shale, much higher than MCIP technique and helium porosimetry results, revealing very high isolated porosity and very low permeability. The findings were investigated and analysed using of extensive FIB/SEM images. These 2D images displayed high amounts of isolated porosity supporting high porosity existence estimated from pyknometry technique. Therefore, it was envisaged that total porosity assessment was not possible by helium porosimetry and mercury injection capillary pressure techniques. The pyknometry technique supported by the SEM images is an alternative method which measures total rather than effective porosity.

From our FIB/SEM experimental work and observations we believe Roseneath and Murteree formations have high level of heterogeneity, anisotropy and possess macro and micro-level natural fracture systems. The observed pores and fractures system strongly support that total porosity is much higher than 2.00% estimated from QEMSCAN, sonic and density porosity from logs data as well as effective porosity from helium porosimetry and MICP techniques.

Later selected water saturation models were applied for water saturation estimation in Murteree shale. Based on cumulative effect and interaction of minerals, fluids and organic content application of Archie’s equation and other resistivity models such as Indonesian, Siamandoux and Total shale model were investigated. Wire-line logs data was calibrated with QEMSCAN, XRD and FIB/SEM images for clay volumes and total porosity. Evaluation of Archie’s parameters (cementation exponent, m, tortuosity factor, a, and saturation exponent, n) were re-examined through sensitivity analysis before applying these models. Using Simandoux and Indonesian models we found 86.00% ($S_w$) and 84.00% ($S_w$) respectively when original porosity 2.00% was considered for Murteree shale. However, when total porosity based on 2D FIB/SEM images was considered to be more than or equal to 10.00%, Archie model gave an estimation of 89.00% and with humble formula 84.00% water saturations respectively. Total shale model estimated 58.00% and 70.00% water saturations when cementation exponent (m) was taken as 2.00 and 2.15 respectively in same Murteree shale interval with a total porosity 10.00%.

# Declaration

I certify that this work contains no material which has been accepted for the award of any other degree or diploma in any university or other tertiary institutions and, to the best of my knowledge and belief, contains no material previously published or written by another person except where due reference has been made in the text. I certify that no part of this work will, in future, be used in submission for any other degree or diploma in any university or other tertiary institution without the prior approval of the University of Adelaide and where applicable, any partner institution responsible for the joint-award of this degree.

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Maqsood Ahmad

Signed: Date: April 14, 2014

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# Dedication

This thesis is dedicated to my parents, brothers, and sister

# Research Activity Flow Chart 2010-2011

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<tr><td>Experimental Techniques & Methodology Selection & Applied:<br>1. Helium Porosimetry<br>2. Mercury Injection Techniques<br>3. Liquid Pyknometry<br>4. QEMSCAN, XRD & FIB/SEM<br>5. Micro and Nano CT-Scanning<br>6. Wireline Logging Techniques<br>Objectives/Tasks:<br>1. Mineral and Rock Characterization<br>2. Effective and Total Porosity<br>3. Pore Throat and Pore Size, Morphology and Micro Fracture System Study and Interconnectivity<br>4. Water Saturation Evaluation</td></tr>
<tr><td rowspan="2">2011</td><td>Wells & Cores Selection (Della#4 and Moomba#46)<br>Samples Collection & Experimental Setup</td></tr>
<tr><td>Sample Preparation for:<br>1. QEMSCAN Radiation<br>2. Micro and Nano CT-Scanning<br>3. XRD – Technique<br>4. SEM Imaging of QEMSCAN samples<br>Experimental Results:<br>1. Organics and Various Minerals Phase Identification & Quantification<br>2. Weight % & Volumetric % of each Mineral Phase<br>3. False Colour Map Preparation of Scanned Samples<br>4. Fractures & Pores Identification in QEMSCAN Used Sample<br>5. XRD – Minerals Identification and Quantification<br>6. Micro and Nano CT-Scanning for Pores and Micro Fracture system<br>Results Correlation:<br>1. QEMSCAN and XRD Results Correlation<br>2. Pores Correlation between FIB/SEM Images and QEMSCAN Results<br>3. Micro – CT Scanning and FIB/SEM Images Correlation for Micro Fracture System</td></tr>
</table>

# Research Activity Flow Chart 2012-2013

| | | |
|---|---|---|
| 2012 | Literature Review | **Sample Preparation for :**<br>**1.** Helium Porosimetry<br>**2.** MICP(Mercury Injection Capillary Pressure)<br>**3.** Liquid Pyknometry<br>**4.** FIB/SEM<br>**Experimental Results:**<br>**1.** Effective Porosity by Helium and MICP<br>**2.** Pores Throat size and Pores Size Description by MICP & FIB/SEM<br>**3.** Total Porosity by Liquid Pyknometry<br>**Results Correlation:**<br>**1.** Helium Porosimetry and MICP Results Correlation<br>**2.** Pores Correlation between MICP and FIB/SEM<br>**3.** Total Porosity from Liquid Pyknometry & FIB/SEM Images & MICP Graphs |
| 2013 | | **Well Completion Reports Selection:**<br>**1.** Della#4 & Wireline Log Data Digitization<br>**2.** Moomba#46 & Wireline Log Data Digitization<br>**3.** Recently drilled Well Completion Reports in Cooper Basins for Murteree and Roseneath Shale<br>**4.** Use of FIB/SEM Images from Murteree & Roseneath Shales<br>**Experimental Results:**<br>**1.** FIB/SEM images Description for Organics and Clays<br>**2.** Fluids, Organics and Mineral Interaction<br>**3.** Resistivity and Conductivity Analysis<br>**4.** a, m, n, $R_w$ and $R_{cl}$ from Published Data<br>**5.** Water Saturation Calculation Using Archie, Indonesian, Simandoux and Total Shale Models<br>**Results Correlation:**<br>**1.** Diagenesis Related Changes in Mineralogy and Fluid Production<br>**2.** Interaction between Clays, Methane and Brine<br>**3.** Interaction Impact on Resistivity<br>**4.** Use of Archie's Formula for Water Saturation and Correlation with Indonesian , Simandoux and Total Shale Model |

**Three research papers were produced and published based on this research as given below with the titles and name of Journal and Conference venues, presented.**

**EVALUATION OF FREE POROSITY IN SHALE GAS RESERVOIRS (ROSENEATH AND MURTEREE FORMATIONS CASE STUDY)**


M. Ahmad, A. Alamar, R.Koo, H. Nguyen and M. Haghighi
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**Mineralogy and Petrophysical Evaluation of Roseneath and Murteree Shale Formations, Cooper Basin, Australia Using QEMSCAN and CT-Scanning**

Maqsood Ahmad, and Manouchehr Haghighi, SPE, Australian School of Petroleum, The University of Adelaide, SA 5005, Australia



This paper was prepared for presentation at the SPE Asia Pacific Oil and Gas Conference and Exhibition held in Perth, Australia, 22–24 October 2012.



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**SPE 167080**

**Water Saturation Evaluation of Murteree and Roseneath Shale Gas Reservoirs, Cooper Basin, Australia Using Wire-line Logs, Focused Ion Beam Milling and Scanning Electron Microscopy**

Maqsood Ahmad, ASP, the University of Adelaide
Manouchehr Haghighi, ASP, the University of Adelaide



This paper was prepared for presentation at the SPE Unconventional Resources Conference and Exhibition-Asia Pacific held in Brisbane, Australia, 11–13 November 2013.



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