

PORE STRUCTURE CHARACTERIZATION OF A LOW PERMEABILITY RESERVOIR USING NUCLEAR MAGNETIC RESONANCE, NITROGEN ADSORPTION AND MERCURY INTRUSION CAPILLARY PRESSURE

Bello Lawal Toro^{1}, Zhiping Li¹, Caspar Daniel Adenutsi¹, Samira Mohammed Abdullahi²*

¹ School of Energy Resources, China University of Geosciences, Beijing, 100083, P.R. China Beijing Key Laboratory of Unconventional Natural Gas Geology Evaluation and Development Engineering, Beijing, 100083, P.R. China

² Walden University 100 Washington Avenue South Minneapolis, Minnesota 55401

Received September 18, 2017; Accepted December 1, 2017

Abstract

Low permeability core is analyzed using a combination of Nuclear magnetic resonance, Nitrogen adsorption, and Mercury injection capillary pressure. The material's surface area and pore size distributions were determined using Brunauer-Emmett-Teller and Barrett-Joyner-Halenda methods, respectively. Results indicate pores in the 2~ 10nm size ranges have a high percentage. The core displayed high irreducible saturation due to the volume of small pores. Mesopore of type V was identified from study carried out. Capillary pressure correlation between nuclear magnetic resonance and mercury injection show good agreement, however discrepancy in total pore volume was observed.

Keywords: Low permeability; Pore size distribution; Nuclear Magnetic Resonance; Nitrogen Adsorption; Mercury Injection Capillary Pressure.

1. Introduction

One of the major challenges engineers face in the development of Oil and gas fields is the characterization of the reservoir. Detailed knowledge of the field translates to a successful recovery operation therefore, reservoir characterization is very vital in enhancing the remaining untapped hydrocarbons in a formation. It helps to identify those crucial elements of the formation which sometimes operate unpredictably. Such elements include the porosity, permeability and relative permeability, pore size distribution and other major factors that will influence production. In essence reservoir characterization is critical in building an integrated reservoir model for conducting a suitable performance analysis and ultimate recovery calculations.

The pore structure characterization refers to the geometry, distribution, size, porosity, specific surface area and characteristics of rock pore. These parameters are crucial in modeling geophysical and petrophysical behavior of any porous media. Recently, more advanced techniques are developed to determine the pore structures characterization. Chalmers *et al.* [1], Curtis *et al.*, [2]; Milliken *et al.*, [3] characterized mudrocks with advanced imaging techniques which reveals a nanometer-scale pore structure within their inorganic and organic components. One limitation to that is the analysis can only provide visual image of the mudrock's porosity but pore-structure profile cannot be obtained directly. Another limitation to that is pores smaller than 5nm cannot be obtained therefore a portion of the pore structure cannot be investigated [4].

Pore characterization is normally estimated by nuclear magnetic resonance (NMR), mercury intrusion capillary pressure (MICP) or nitrogen adsorption-desorption method (N₂). Consequently, the pore size distribution can be used to examine the fluid flow characteristics of reservoir rocks.

Most researchers use either one or two methods to analyze and characterize the results. Such as Shimokawara *et al.* [5] characterized the pore size distribution by mercury injection

and NMR T_2 relaxation time distribution; Jamal Hassan [6] used the nitrogen adsorption-desorption and NMR technique, to determine the pore size distribution of nano-silica material MCM-41; Sørland *et al.*, [7] used the NMR technique to determine the pore size distribution of core samples. In this study a combination of the three methods to characterize a low permeability rock.

2. Methodology

For the purpose of this entire study, sample of a core was collected for a low permeability reservoir. The sample is in a cylindrical shape. Prior to the NMR experiment the sample was fully saturated in brine for 48 hours under a pressure of 30 MPa. The high pressure was selected because of the very low permeability nature of the sample

2.1. Nuclear magnetic resonance (NMR)

The NMR laboratory measurement was performed using a SPEC PMR machine. The machine operates at a resonance frequency of 21.89 MHz. The measurement was done at echo numbers of 1024 and environment temperature of 32°C. Other parameters are scanning numbers of 64; waiting time is 1s and echo spacing of 1ms. The brine saturated sample was wrapped in a plastic wrap and placed in the machine for the test. In NMR analysis test, samples are scanned when fully saturated with a fluid. This will give the pore size distribution of the sample represented by T_2 . NMR tools are also used to determine the petrophysical properties of reservoir rocks such as permeability, porosity, irreducible water saturation etc. This is a fast and non destructive technique that analyzes the chemical and physical properties of a material. Normally, the relaxation rate $1/T_2$ is proportional to the surface to volume ratio of the pore space and the surface relaxivity in a porous system given in equation 1.

$$\frac{1}{T_2} = \rho_2 \left(\frac{S}{V} \right) \quad (1)$$

where the relaxation rate $1/T_2$ is in 1/ms, surface to volume S/V is in $(1/\mu\text{m})$ and the surface relaxivity ρ_2 is in $(\mu\text{m/s})$.

2.2. Mercury injection capillary pressure

Before the mercury injection experiment was carried out, the sample was dried in an oven for 48 hours, at 110°C to remove all brine from the interconnected pores. Mercury injection test was performed by Quantachrome Poremaster automatic pore size analyzer. The sample was weighed and placed into a penetrometer and loaded into the pressure chamber of the machine. The equipment is capable of injecting mercury through the penetrometer into a dried core plug or cut sample with incrementally increasing the pressure up to 33,000psi (227MPa). The volume of the injected mercury at each pressure increment is recorded until the maximum pore volume mercury saturation is achieved. The pressure is plotted against incremental mercury saturation to achieve a drainage curve. This process can be reversed to generate a non-wetting phase imbibitions curve. Mercury porosimetry is based on the capillary law governing liquid penetration into small pores. Capillary forces in the reservoir and seal are functions of surface and interfacial liquid tensions, pore-throat size and shape, and the wetting properties of the rock. The pore throat radius can be determined by Washburn equation:

$$r = \frac{2\sigma\cos\theta}{P_c} \quad (2)$$

where r is the pore throat radius; θ is the contact angle; σ is the interfacial tension (dynes/cm) and P_c is the capillary pressure.

2.3. Nitrogen adsorption/desorption method

The machine used in the study is a specific surface and pore size analysis instrument, manufactured by Beishide instrument technology Beijing Co. Ltd. This instrument works upon the principle of physical adsorption and has provision to use a few gases, viz. nitrogen, argon, krypton, as adsorbate on various adsorbents.

The experiment was conducted using the same core that was used in the NMR and MICP experiment. The sample was dried in an oven at 110°C for 48 hours. The sample was then

crushed using minimal energy to pass through a sieve. Approximately 4g of the material was used for the test. This is based on the methodology proposed by McCarty [9] because it is effective in producing mineralogically and chemically homogenous splits. Prior to the start of the adsorption process, the sample was degassed under vacuum to remove any unwanted vapors and gases adsorbed on the sample surface. The temperature for degassing was set to 200°C.

3. Results

3.1. NMR

NMR tools provide information on the amount of fluids present in cores, and also the size of pores that are filled with the fluids. This is one of the features that distinguish it from other logging devices. The NMR pore size distribution for the core sample is shown in figure 1. The result of the T_2 spectrum shows a bi-modal distribution showing the T_2 distribution of all pores in the core sample representing the pore size distribution. The population is situated around 1.86ms. The NMR spectrum clearly shows one peak much larger in space and amplitude. The area covered by the larger peak is the BVI (Bulk volume immoveable) with a massive proportion of about 82% of Irreducible water saturation (Swirr) leaving a low proportion for Free fluid index (FFI). This indicates that there are poor pore size distributions within the core.

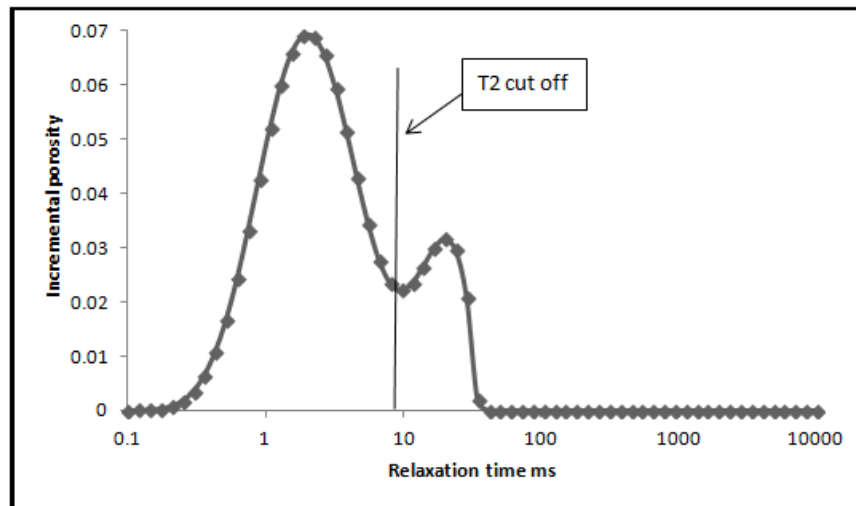


Figure 1. NMR spectrum for sample

The NMR test was able to measure and identify the fully saturated and bound fluid. Results show that the core consists of large proportion of micropore body which is typical of low permeability reservoirs. The spectrum reveals that large proportion of the total volume consist of small pores characterized by small matrix grains. This will cause high irreducible water saturation (Swirr) in the formation. In order to improve the reliability of the formation, it is imperative to measure the Swirr. Determining Swirr on the NMR spectrum requires a T_2 cutoff mark to be identified. The T_2 cutoff mark will define the proportion of Bulk Volume Irreducible (BVI), and Bulk Volume Moveable (BVM) this is also known as Free Fluid Index (FFI). The standard T_2 cutoff for sandstone reservoirs is 22ms [10]. In low permeability sandstones and shale gas fields, the T_2 cutoff mark is around 10msec. The traditional clay bound cut value of 2.5msec are typically too low as well. A value around 1ms is more representative for these types of rocks [11]. This is a common method for determining the Swirr from the NMR by identifying the T_2 cutoff on the log [12].

3.1.1. Estimating permeability from NMR logs

Different empirical equations are suggested for estimating permeability of a porous media using NMR. Many authors presented works on estimation of permeability from NMR [13-16]. However, Coates model [17] is the most widely used. This model can be applied on formations containing water and/or hydrocarbons. The Coates model is used in computing permeability [14]:

$$K = \left[\left(\frac{\phi}{C} \right)^2 \left(\frac{FFI}{BVI} \right) \right]^2 \quad (3)$$

where, k = permeability; with ϕ for total porosity (%); C=10 or can be determined from laboratory measurements on cores; FFI= the free fluid index and BVI = the bulk volume of irreducible water.

Rezaee *et al.* [18] did a study on some cores and came up with a new correlation for estimating permeability. They proposed that using NMR T₂ dominant peak or T₂ peak and multi-regression analysis, permeability can be estimated with high accuracy. The multi-regression analysis resulted in the following equation for permeability estimation using T₂ peak and porosity:

$$K = -0.0461 - 0.0601T_{2peak} + 4.37\phi \quad (4)$$

where k is permeability (mD), T_{2peak} is dominant T₂ on the T₂ spectrum (ms), and ϕ is porosity (fraction). Using this equation, permeability was estimated to be 0.2mD.

3.1.2. Estimating pseudo-capillary pressure curves using NMR logs

Research has been and it is still carried out on how to manipulate the NMR spectrum to determine the properties of hydrocarbon formation and give a proper evaluation. Volokitin *et al.* [19] was among the pioneers of researching how to convert NMR spectrum to pseudo-capillary pressure curve. Later on so many research was carried out on using the logs to derive the pseudo Pc curve [20-23]. Volokitin *et al.* [19] method is commonly used in the characterizing the pore structure. However, Liang Xiao *et al.* [23] carried out test using the same method and discovered that the method is not applicable on the formation in China and tight sandstone reservoirs. They also stated that the conventional linear function proposed cannot be used adequately to predict Pc curves from NMR logs. They proposed a whole new method based on formation classification in their research named Classified Piecewise Power Function (CPPF). In their research the NMR reverse cumulative curves was used to construct the PC curves (Details of the procedure is explained in their paper). The capillary pressure using NMR reverse cumulative curve is calculated as proposed by Liang Xiao *et al.* [23] using the CPPF method. The curve is correlated to that of MICP for validation as illustrated in figure 2b. Good agreement was obtained from the capillary pressure measured from the two methods. This further shows a good relationship between the two experimental methods.

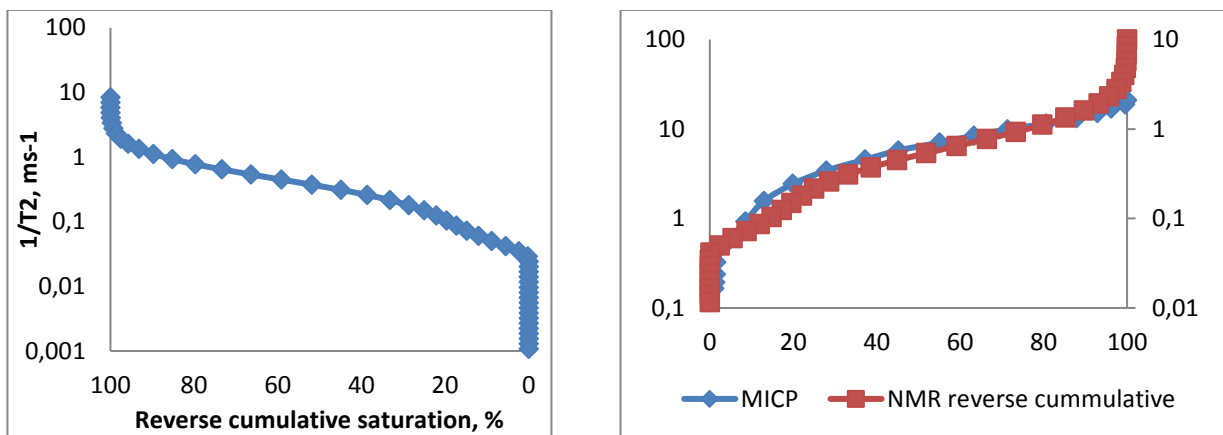


Figure 2. (a) NMR reverse cumulative curve

(b) Pc curve comparison between MICP and NMR

3.2 MICP

Mercury was injected at a high pressure of 10,000psi on the sample. Drainage capillary pressure and pore throat distribution were produced as shown in figure 3. Due to the low permeability nature of the core, the capillary pressure is high. The minimum pressure (P_d), also known as the displacement pressure, the threshold pressure is determined by the size of the largest pores connected to the surface of the medium [24]. In this case the entry pressure is 0.52mPa. The inflection point corresponds to 9.19 mPa at 8.52 saturation of mercury. The Washburn equation (Equation 2) was used to convert capillary pressure into estimated throat radius. Pore throat distribution curve (figure 3b) suggest significant pore volume in micro pores with a bi modal PSD for the sample. Dan J. Hartman and Edward A. Beaumont [25] classified pore sizes as nanopores < 0.1 μm , micropores 0.1-0.5 μm , mesopores 0.5-2 μm , macropores 2-10 μm and megapores > 10 μm . Majority of the pore throat sizes fall between 0.1-1 μm indicating that the core falls within the range of micropores and mesopores. It should be noted that mercury injection experiments doesn't measure all pore throats but only throat accessible to the mercury.

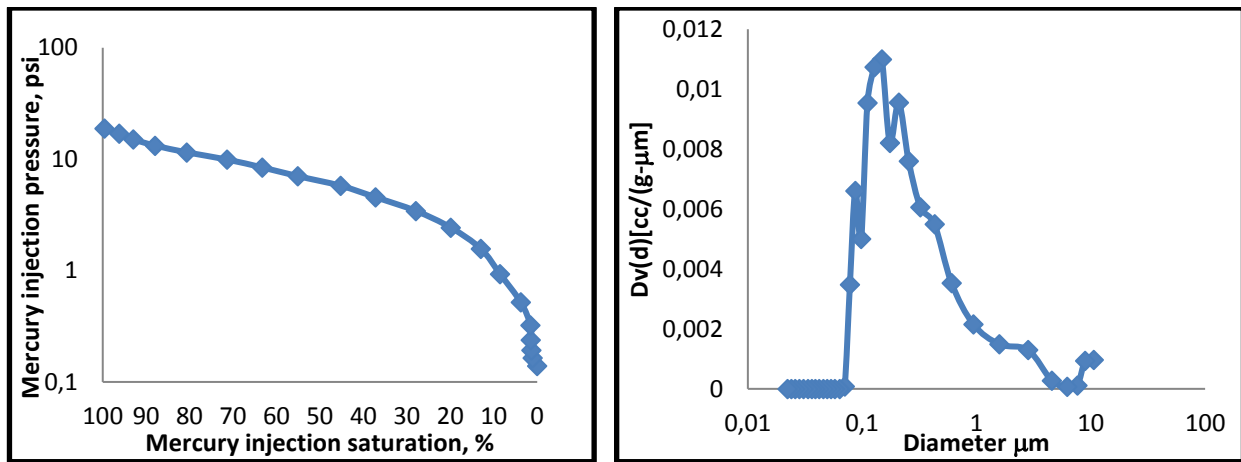


Figure 3. (a) Capillary pressure curve

(b) Pore throat distribution

3.2.1. Relative permeability

Relative permeability is of central importance to soil science, petroleum engineering, and many other industries but may be difficult to measure in some cases. Such cases include extremely low permeability rocks and special fluid systems in which there are phase transformation and mass transfer between the two phases as pressure changes [26]. There are different studies done on computing relative permeability from capillary pressure to [27-30]. Brooks and Corey's model for calculating relative permeability is a method widely used for calculating relative permeability. Brooks and Corey [31] modified Corey's capillary pressure model (equation (5)) and presented a new capillary pressure function model based on evaluations from several drainage capillary pressure curves from consolidated porous media as follows:

$$P_c = P_e(S_w^*)^{-\frac{1}{\lambda}} \tag{4}$$

where: λ pore size distribution index; P_e = entry capillary pressure; P_c = capillary pressure as a function of S_w .

Finally, they derived equation (6) and equation (7) to calculate the true relative permeability curve for the wetting and non-wetting phase.

$$K_{rw} = (S_w^*)^{\frac{2+3\lambda}{\lambda}} \tag{5}$$

$$K_{rnw} = (1 - S_w^*)^2 \left[1 - (S_w^*)^{\frac{2+\lambda}{\lambda}} \right] \tag{6}$$

where: K_{rw} is the relative permeability of the wetting phase; K_{rnw} is the non-wetting phase relative permeability at the irreducible wetting phase saturation; S_w^* is the normalized wetting phase saturation, which could be expressed as follows:

$$S_w^* = \frac{S_w - S_{wi}}{1 - S_{wi}} \quad (7)$$

where: S_{wi} is the irreducible wetting phase saturation.

In this study the Brooks and Corey [31] relative permeability model is used to calculate relative permeability from the capillary pressure data and the result is presented in figure 4.

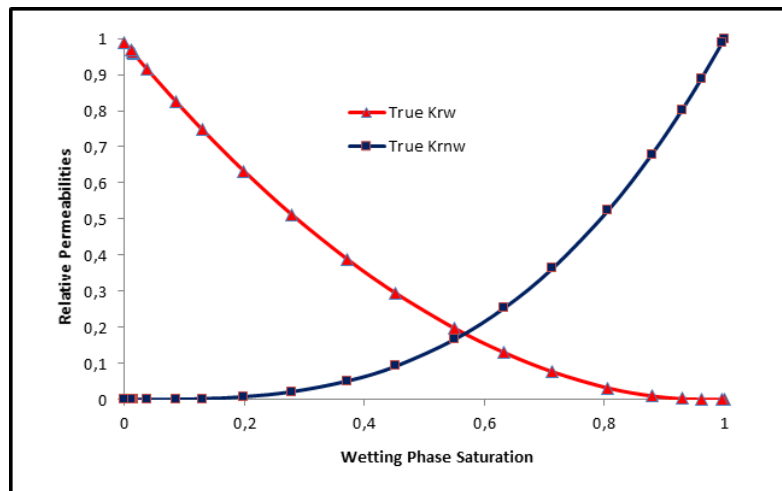


Figure 4. Relative permeability curves

3.3. Nitrogen adsorption/desorption method

The BET (Brunauer, Emmet and Teller) method was used to calculate the surface area of the samples. This technique is universally employed for determining surface area of porous materials because of its simplicity, its definitiveness and its ability to accommodate each of the five isotherm types [32].

Isotherm is the first significant information that is obtained from a physisorption experiment about surface area and porosity of porous materials. During the BET test, there is capillary condensation of liquid nitrogen around the pores and computation of the amount of nitrogen absorbed at a given relative pressure of the sorption isotherm.

The adsorption and desorption isotherms for the sample is shown on figure 5. During the adsorption isotherm process, the monolayer adsorption is formed at low relative pressure. At high relative pressure, the adsorption in mesopores will cause the multilayer formation until the capillary condensation occurs. As the relative pressure starts to increase there's gradual increase of adsorption as well. With the increase in relative pressure the pores are filled with nitrogen and condense. This causes the diameter of the pore filled with the nitrogen to increase gradually. The results shows the adsorption/desorption curves are smooth and give a good hysteresis showing a capillary condensation transition. Based on the IUPAC classification of sorption isotherms by Brunauer, Demming, Deming, and Teller (BDDT), the curves suggest that of type V. The curves show pore condensation and hysteresis which closes at relative pressure of 0.4-0.45 P/Po. As relative pressure reaches 0.98, the core sample absorbed the highest volume of N₂ of 4.36ml/g. The surface area is 1.080m²/g. The curves indicate weak attractive interactions between the adsorbate and adsorbent.

The BJH (Barret, Joyner and Halenda) method was used to determine the pore size distribution of the samples. This method of calculating pore size distribution is from the experimental isotherms using the Kelvin model of pore filling. The BJH computation method starts from higher pressures, hence larger pore sizes, with lower pressures (smaller pore sizes)

in the later steps. Figures 6 show the curves of the pore size distribution in the process of the BJH adsorption and desorption. According to the pore size distribution curves of the BJH, the pores are mainly distributed and centered between 2-10nm. Therefore, according to the classification of pores by the International Union of Pure and Applied Chemistry (IUPAC), the samples are classified as mesopores. The total pore volume determined by BJH is 0.0068 ml/g.

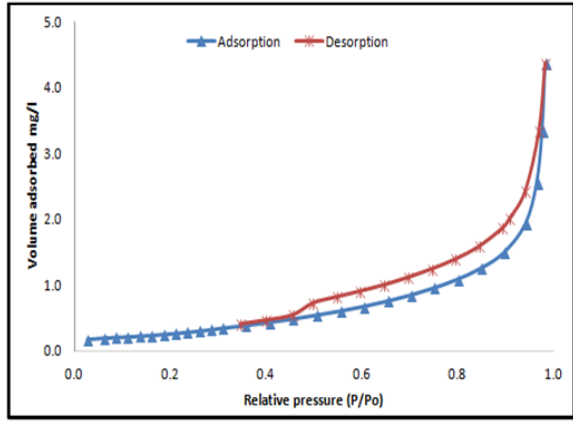


Fig. 5. Adsorption/desorption isotherm for sample

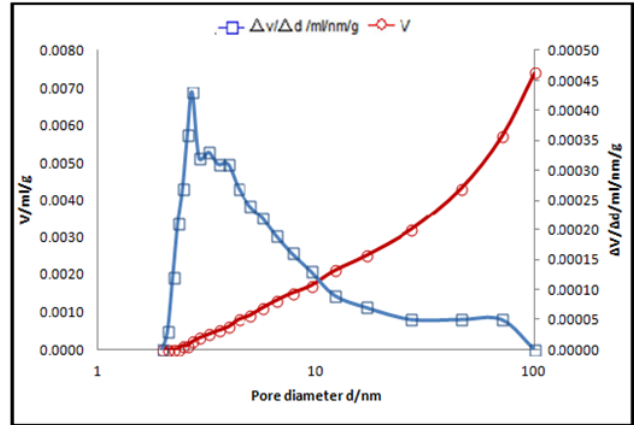


Fig. 6. BJH pore size distribution for sample

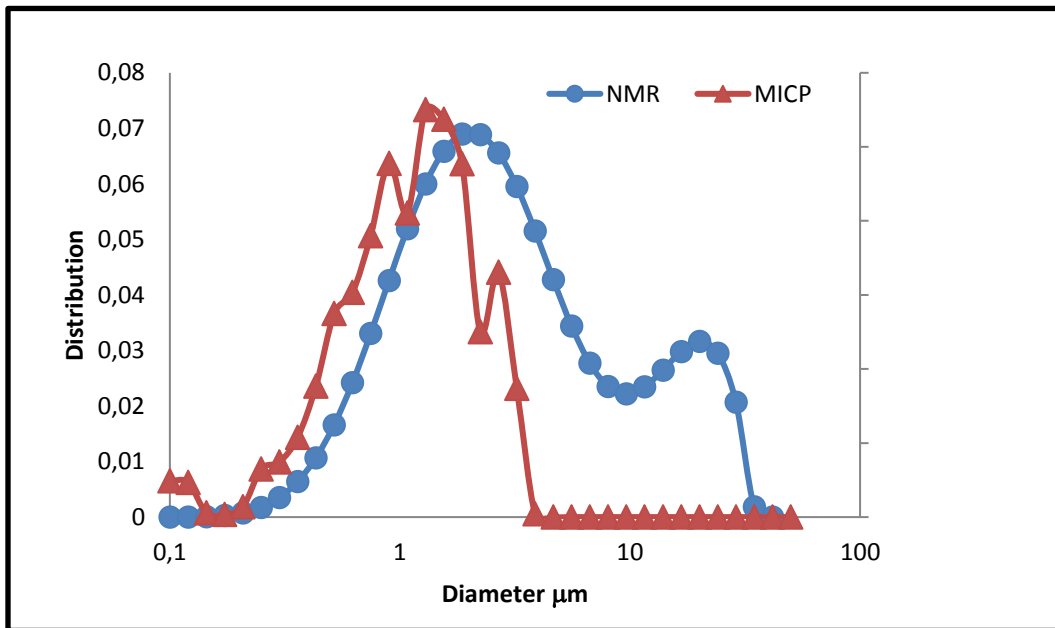


Figure 7. PSD correlations between NMR and MICP

4. Discussion

MICP has a limited disadvantage compared to the N_2 . Its pore diameter range is from 3nm and above, while that of N_2 has the ability to measure pores that are less. However, the MICP covers higher range pore diameters measurement compared to N_2 . For materials containing large pores, the MICP is a preferred technique. Due to its high pressure ability, it is suitable for core of high, medium and low permeability. However, in most cases the mercury injection pressure is not high enough to access small pores. Estimating pore diameters greater than 350nm are rarely used from the gas sorption method. The operational procedures for MICP and N_2 is different while the former first measures larger pores at intrusion phase and the latter first measure smallest pores at the adsorption phase. Surface area of the sample

estimated by MICP is lower than the N_2 . According to Milburn and Davis [33] correlation between surface area obtained from MICP and N_2 is poor if the samples have low surface area. The combination of these three methods for pore characterization is an effective way for understanding the petrophysical properties of the low permeability reservoirs.

Pore size distribution for NMR and MICP are correlated and shown in figure 7. There is a fair agreement between the spectrums. When the PSD curves are fitted, they overlay the distribution with a matching peak. They also show a similar distribution. Experimental work on NMR is much faster than the other methods followed by MICP. Total pore volume calculated by NMR is a bit higher than that measured by N_2 . This may be due to the reason that NMR measures both dead end pores with only one entry to the main pore channel and the interconnected pores that support the flow of fluids.

5. Conclusion

In this study pore structure characterization of a low permeability core was reviewed using Nuclear Magnetic Resonance (NMR), Mercury injection capillary pressure (MICP), and Nitrogen adsorption (N_2).

Results for the permeability, total pore volume, surface area, were obtained. Pore throat distribution using MICP indicate dominant of the pores lie within 0.1-1 μ m making them fall within the micro and mesopore range. Capillary pressure curve of MICP and NMR was correlated and show good agreement. However, there was discrepancy of Total pore volume between NMR and N_2 . Type V mesopore of IUPAC classification of sorption isotherm was identified using the N_2 adsorption method.

Relative permeability was obtained using Brooks and Corey method. Ideally, low permeability cores should have low flow rates. By applying low flow rates to the core, some difficulties are likely to occur like low pressure changes which are intricate to measure and long duration of experiments which in some cases might take days. Therefore using the Brooks Corey method will be less time consuming and less complex.

Acknowledgement

The research was supported by Beijing Municipal Natural Science Foundation (grant number: 3162026). The authors wish to express their sincere appreciation to Dr. Xiao Liang for his guidance and contributions. We would also like to thank Xu Zhi Chao (Jerry) for his assistance in the lab. The authors also thank the reviewers of the manuscript.

Reference

- [1] Chalmers, G. R., R. M. Bustin, and I. M. Power, 2012. Characterization of gas shale pore systems by porosimetry, pycnometry, surface area, and field emission scanning electron microscopy/ transmission electron microscopy image analyses: Examples from the Barnett, Woodford, Haynesville, Marcellus, and Doig units: AAPG Bulletin, 96, no. 6, 1099–1119.
- [2] Curtis, M. E., B. J. Cardott, C. H. Sondergeld, and C. S. Rai, 2012. Development of organic porosity in the Woodford Shale with increasing thermal maturity: International Journal of Coal Geology, 103, 26–31.
- [3] Milliken, K. L., M. Rudnicki, D. N. Awwiller, and T. Zhang, 2013. Organic matter-hosted pore system, Marcellus Formation (Devonian), Pennsylvania: AAPG Bulletin, 97, no. 2, 177–200.
- [4] Kuila U and Prasad M. Colorado School of Mines. Application of nitrogen gas-adsorption technique for characterization of pore structure of mudrocks, December 2013
- [5] Shimokawara M, Kaido H, Mino Y. 2011. Application of NMR for Special Core analysis, iea-eor.ptrc.ca/2011/assets/25_18-10-2011_15-50_Paper.pdf.
- [6] Hassan, Jamal 2012. Pore size distribution calculation from 1H NMR signal and N_2 adsorption desorption techniques, Physica B condensed Matter.
- [7] G.H. Sørland, K. Djurhuus, H. C. Widerøe, J.R. Lien, and A. Skauge. 2007. Absolute pore size distributions from NMR.
- [8] Daniel R, and Kaldi J. 6/8/12. Mercury-injection capillary-pressure analysis store-assets.aapg.org/documents/previews/1044ST60/CHAPTER01.pdf

- [9] McCarty DK. 2002 Quantitative mineral analysis of clay-bearing mixtures. The Reynolds cup contest: IUCr CPD Newsletter, 2002; 27: 12-16.
- [10] Dunn KJ, LaTorraca GA, Warner JL, Bergman DJ. On the Calculation and Interpretation of NMR Relaxation Time Distributions, SPE 69th Annual Technical Conference and Exhibition, New Orleans 1994, SPE28367
- [11] Green DP and Veselinovic D. Analysis of Unconventional Reservoirs using New and Existing NMR Methods, 2010
- [12] Xiao L, Mao Zh, Jin Y. Calculation of Irreducible Water Saturation (Swirr) from NMR Logs in Tight Gas Sands. November 2011
- [13] Kleinberg, R.L., 1996. Utility of NMR T2 distributions, connection with capillary pressure, clay effect, and determination of the surface relaxivity parameter ρ_{02} . Magn. Reson. Imaging 14, 761-767.
- [14] Howard, j., Williams, J., Thorpe, D. 1997. Permeability from nuclear magnetic resonance logging in a gas-condensate field. SPWLA 38th Annual Logging Symposium Transactions.
- [15] Epping, W.j., Eggenkamp, I.M., Reid, I. 1999. Added Value from NMR Measurements to Characterize Gas Reservoirs in the UK Southern North Sea.
- [16] Glove, PW, Zadjali LL, Frew KA. Permeability prediction from MICP and NMR data using an electrokinetic approach, Geophysics, 2006; 71(4): F49-F60.
- [17] Coates GR, Xiao L, Prammer MG. 1999. NMR Logging – Principles and Applications. Halliburton Energy Services, Houston;
- [18] Rezaee R, Saeedi A, Clennell B. December 2011. Tight gas sands permeability estimation from mercury injection capillary pressure and nuclear magnetic resonance data.
- [19] Volokitin Y, Looyestijn WJ, Slijkerman W.J, Hofman JP. A practical approach to obtain 1st drainage capillary pressure curves from NMR core and log data. Paper SCA-9924 presented at the SCA annual symposium 1999.
- [20] Ben Lowden, Res Lab-ART, Suffolk, May 2000. Some simple methods for refining permeability estimates from NMR logs and generating capillary pressure curves.
- [21] Grattoni CA. et al. September 2003. An Improved Technique for Deriving Drainage Capillary Pressure from NMR T2 Distributions. Presented at the International Symposium of the Society of Core Analysts, Pau, France. SCA.
- [22] Shao, W.Z.; Ding, Y.J.; Liu, Y; Liu, S.Q.; Li, Y.Q.; Zhao, J.H. 2009: The application of NMR log data in evaluation of reservoir pore structure. Well Logging Technol. 33(1), 52–56.
- [23] Liang Xiao, Zhi-qiang Mao, Chang-chun Zou, Yan Jin, Ji-chang Zhu 2016. A new methodology of constructing pseudo capillary pressure (P_c) curves from nuclear magnetic resonance (NMR) logs.
- [24] Ekwere J. Peters. 2012. Advanced Petrophysics: Volume 2: Dispersion, Interfacial Phenomena/Wettability, Capillarity/Capillary Pressure, Relative Permeability.
- [25] Dan J. Hartman and Edward A. 1999. Beaumont Exploring for oil and gas traps.
- [26] Kewen Li and Roland N. Horne 1st Feb, 2006. Comparison of methods to calculate relative permeability from capillary pressure in consolidated water-wet porous media.
- [27] Purcell WR. 1949. Capillary pressures—Their measurement using mercury and the calculation of permeability, Trans. AIME, 186, 39.
- [28] Burdine NT. Relative permeability calculations from pore size distribution data. Trans. AIME, 1953; 198, 71–78.
- [29] Lenhard, R.J., Oostrom, M., A 1998. parametric method for predicting relative permeability saturation–capillary pressure relationships of oil–water systems in porous media with mixed wettability. Transp. Porous Media 31 (1), 109.
- [30] Li, K., Horne, R.N. 2001. An experimental and theoretical study of steam–water capillary pressure. SPEREE 477–482.
- [31] Brooks RH, Corey AT. Properties of porous media affecting fluid flow. J. Irrig. Drain. Div. 1966; 6, 61.
- [32] Lowell S., Shields JE, Thomas MA and Thommes M. Characterization of porous solids and powders: Surface area, pore size and density, Springer 2006.
- [33] Milburn DR and Davis BH. 1993. Comparison of Surface Areas Calculated from Nitrogen Adsorption and Mercury Porosimetry, in A Collection of Papers on Engineering Aspects of Fabrication of Ceramics: Ceramic Engineering and Science Proceedings, 1993; 14(11/12) (ed J. B. Wachtman), John Wiley & Sons, Inc., Hoboken, NJ, USA. doi: 10.1002/9780470314272.ch8.

To whom correspondence should be addressed: Dr. Bello Lawal Toro, School of Energy Resources, China University of Geosciences, Beijing, 100083, P.R. China, bltoro@yahoo.com