

Study on high pressure imbibition characteristics of ultra-deep carbonate rocks based on nuclear magnetic resonance method

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ABSTRACT

In dengsi gas reservoir, Anyue gas field, Sichuan Basin, It's diagenetic process of ultra-deep carbonate sedimentation is complex. At present, the structural characteristics of the pore throat are not enough. In this paper, I use the real reservoir core of the gas reservoir as the research object, and study the types and characteristics of the pore throat of the reservoir by using the method of cast thin section analysis and nuclear magnetic resonance testing. Studying the nuclear magnetic resonance spectroscopy characteristics of the three types of pore cores after different pressure imbibition of saturated water. The results show that the main peaks of T2 spectrum of pore type I, II and III cores are distributed around 1-10ms, 10-100ms and 1ms, and the high-speed conductivity of fractures can improve the overall imbibition degree of pore type II cores. Increasing saturation pressure has the greatest effect on the imbibition saturation degree of pore type II cores, and the best effect on the cores with permeability between 0.001-0.01mD. Because it is difficult for water to enter the pore type III core with too low permeability, the influence of increasing saturation pressure on the pore type III core is low. The research can provide a theoretical basis for the study of water invasion mechanism in the production process of target gas reservoir and the drainage gas production in the later stage.

Keywords: Carbonate rock, Nuclear magnetic resonance, Imbibition

1. INTRODUCTION

Ultra-deep Marine carbonate rocks are extremely complex and special reservoirs. Due to the frequent rise and fall of sea level and the influence of fresh water dissolution in the process of sedimentation and diagenesis¹⁻³, the pore throat structure and fluid

distribution of the reservoir rocks are very complicated, and the core is highly dense and heterogeneous⁴⁻⁶. At present, the pore throat structure characteristics of ultra-deep carbonate rocks are commonly analyzed by means of cast thin slice, scanning electron microscope and nuclear magnetic resonance, but static pore throat structure is generally described. There are few studies on the imbibition phenomenon in relatively dense carbonate rock cores and the influence of imbibition effect of pore throat structure. Therefore, it is necessary to conduct in-depth research on imbibition phenomenon in ultra-deep carbonate rock cores.

This paper provides theoretical guidance for recognizing the pore structure characteristics of Deng4 gas reservoir and the changes of gas and water occurrence in the development process by analyzing the subject thin section images of different types of ultra-deep carbonate rock and the NMR spectra characteristics under different imbibition pressures.

2. EXPERIMENTAL MATERIALS AND METHODS

2.1 Experimental core

In this paper, the real reservoir core of Dengsi gas reservoir in Anyue Gas field, Sichuan Basin is used. In order to study the imbibition of formation water in tight core during gas reservoir exploitation, the core is a plunger core with a diameter of 25mm and the core permeability range is 0.0003-0.132mD. Small rock samples generated during core drilling are used to conduct the cast thin section experiment. Detailed core parameters are shown in Tab. 1 below.

Tab. 1. Experimental core parameters

Sample number	Diameter /mm	Length /mm	Porosity /%	Permeability /mD	Core type
GNZ-1	25.46	40.17	6.45	0.113	Porous Core I
GNZ-2	25.28	33.66	5.47	0.132	Porous Core I
GNZ-3	23.74	44.57	8.49	0.0088	Porous Core II
GNZ-4	25.02	40.13	9.36	0.008	Porous Core II
GNZ-5	25.52	40.68	4.96	0.0003	Porous Core III
GNZ-6	23.65	38.58	5.51	0.0006	Porous Core III

2.2 Experimental Instruments

ZTHJ-3A type pore casting instrument and SPEC-RC2 high temperature pressure displacement NMR online displacement analyzer were used in the experiment.



Fig. 1. ZTHJ-3A type rock pore casting instrument



Fig. 2. SPEC-RC2 NMR online displacement

2.3 Experimental methods

Provide sufficient detail to allow the work to be reproduced. Methods already published should be indicated by a reference: only relevant modifications should be described.

In the research process of this paper, two experimental methods are mainly used: cast wafer experiment and nuclear magnetic resonance test. The

specific experimental conditions and processes are as follows:

(1) Analysis method of thin section of cast body: ① The cut sample is manually trimmed to a size that can be placed in the cast tube, separated by mica in the middle, and vacuumed for 2 hours; ② With the already prepared methyl blue and epoxy resin and (1.5%) mixture, add triethanolamine, the proportion of triethanolamine is 15%, pumped into the casting tube, and pumped for about 20 minutes, put into the casting machine; ③ Pressure heating for 4-6 hours, the temperature is 65 degrees, the pressure is 50MPa, and then it is heated to 95 degrees, the pressure is 50MPa, and it is kept for 20 hours; ④ After the temperature drops to room temperature, remove the cast tube from the casting machine, smash the cast tube, separate each sample on the slicer, and grind according to the requirements for grinding flakes; ⑤ Casting image production: slide size, 25.4*76.2mm. After pressing the casting glue into the sheet at high temperature and high pressure, ground the flat surface and stick it on the slide, grind it into 0.035mm thickness, cover the slide with 24*24mm thick 0.17mm, clean it and label it.

(2) High pressure imbibition NMR experimental procedure: ① Core preparation, core washing, drying, porosity measurement and gas permeability measurement; ② Saturated formation water, the core is completely saturated with formation water for 12h by vacuum saturation method, and nuclear magnetic resonance test is carried out after saturation; ③ The core after the completion of NMR test was put into a high pressure saturation kettle for imbibition experiment for 24h, and then NMR test was performed; ④ The pressure of the high pressure saturated kettle is gradually increased, and the experiment of step ③ is repeated.

3. EXPERIMENTAL MATERIALS AND METHODS

3.1 Results of the cast wafer test

According to the thin slice image of rock casting, the reservoir space of the target block mainly contains small size karst cave, solution hole and intergranular hole, and the seepage channel mainly contains cracks and intergranular throat. Cracks are used as percolation channels in some karst caves and large solution pores.

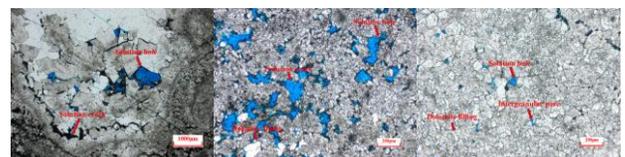


Fig.3 . Flake image of core cast

Throat is the main percolation channel in some solution pores. Throat is generally used as the main percolation channel in intergranular pores. Some of the dissolved pores were filled with dolomite in the process of sedimentary diagenesis.

3.2 Results and analysis of NMR experiments

3.2.1. Pore type I core analysis

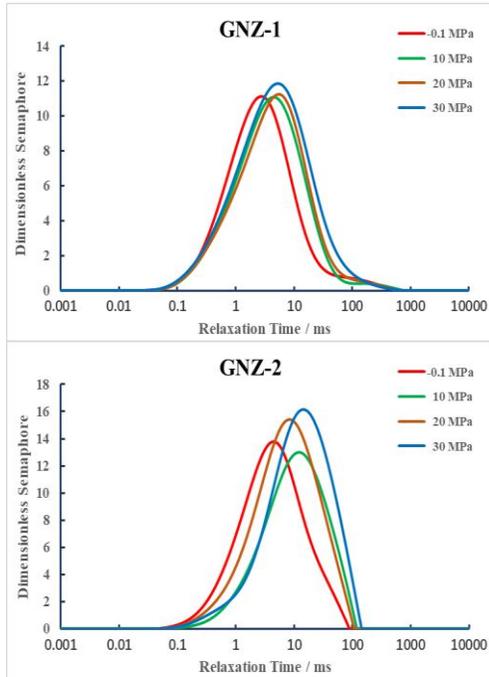


Fig.4 . Different saturation pressure dialyzing of type I

As can be seen from Fig. 4., the T2 spectrum of pore type I core GNZ-1 and GNZ-2 has the same change characteristics with the continuous increase of saturation pressure. Under the vacuum saturation condition, the main peak of pore type I is distributed between 1 and 10ms. When the saturation pressure rises to 10MPa, the imbibition phenomenon of part of the throat is strengthened, the water saturation of connected pores increases, the relaxation time of these pores increases, and the T2 spectrum shifts to the right. When the saturation pressure increases to 20MPa and 30MPa, the imbibition effect of small holes will be enhanced, and the saturation of some pores will be further increased, and the signal volume of T2 spectrum will further increase, and the spectrum will shift upward. Cracks play a very important role in the process of porosity type I core saturation. On the one hand, the existence of cracks can directly communicate with some pores, so that water can directly enter these pores through cracks; on the other hand, the existence of cracks as hyperpermeability channels can greatly reduce the seepage resistance of water, so as to communicate

more seepage throats. Therefore, in the stage of core vacuum saturation, the imbibition effect is the most obvious and the saturation degree is the highest due to the existence of cracks in pore type I core.

According to the characteristics of nuclear magnetic

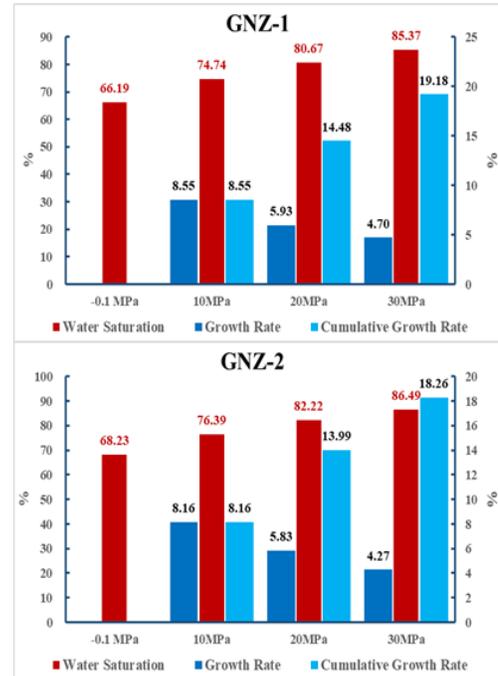


Fig.5 . Different saturation pressure dialyzing of type I

resonance spectroscopy and weighing method, the saturation degree under different saturation pressures is calculated, and the core imbibition characteristics under different pressures are compared. As shown in Fig. 5., the vacuum saturation of pore type I core is relatively high, and GNZ-1 and GNZ-2 are 66.19% and 68.23%, respectively. The high permeability of the core and the development of internal micro-fractures will reduce the resistance of water flow into the core. With the continuous increase of saturation pressure, the increase amplitude of water saturation of GNZ-1 and GNZ-2 decreased from 8.55% and 8.16% to 4.70 and 4.27%, respectively, and the increase amplitude of cumulative water saturation was 19.18% and 18.26%, respectively. With the increasing of saturation pressure, the imbibition phenomenon in the core becomes weaker and weaker. On the one hand, the solution pore connected to the imbibition throat has been completely saturated; on the other hand, the reservoir space of the small pore throat requiring higher pressure to significantly enhance its imbibition effect is small.

3.2.2. Pore type II core analysis

The permeability of GNZ-3 and GNZ-4 cores used in the experiment are 0.0088mD and 0.008mD, and the

porosity is 8.49% and 9.36%.

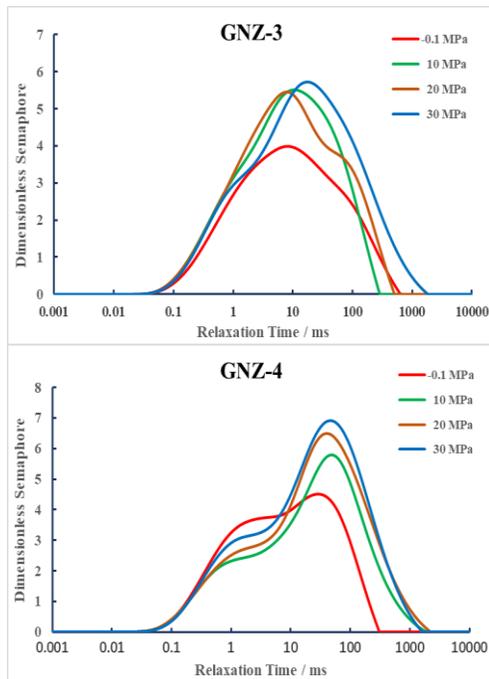


Fig.6 . Different saturation pressures of type II cores

As can be seen from Fig. 6., the change characteristics of T2 spectrum of pore type II core GNZ-3 and GNZ-4 are not exactly the same as those of pore type I core with the continuous increase of imbibition pressure. Under the condition of vacuum saturation, the main peak of pore type II is distributed in the range of 10-100ms, and when the saturation pressure rises to 10MPa, it is the same as that of pore type I . The imbibition phenomenon of some throats is strengthened, more water enters the connected pores, the semaphores increase, and the T2 spectrum shifts to the upper right. This process is mainly due to the increase in the saturation degree of unsaturated pores and the activation of some new throats. When the saturation pressure rises to 20MPa and 30MPa, saturated water gradually intrudes into the core along the throat, which will enhance the imbibition effect of small holes, further increase the saturation of some pores, further increase the signal volume of T2 spectrum, and shift the spectrum up. This process is mainly due to the start-up of new pore throat, the overall upward shift of T2 spectrum, the overall increase of saturation degree, and the increase of saturation pressure has a high degree of influence on pore type II core.

As can be seen from Fig. 7., compared with pore type I core, the vacuum saturation of pore type II core is somewhat reduced, and GNZ-3 and GNZ-4 are 49.85% and 47.51%, respectively. Pore type II core mainly relies on throat as the main seepage channel, and water

cannot enter the core depth during the vacuum saturation process, resulting in limited imbibition. With the continuous increase of saturation pressure, although new pore-throats were used continuously, the number of new pore-throats used became less and less. The increase rate of water saturation of GNZ-3 and GNZ-4 decreased from 12.80% and 13.24% to 6.30% and 6.23%, respectively, and the increase rate of cumulative water saturation was 29.37% and 28.94%, respectively. The vacuum saturation of pore type II core is lower than that of pore type I core, but the influence of increasing saturation pressure on core saturation is greater than that of pore type I core. Imbibition mainly occurs in small throat. Due to the existence of cracks in pore type I core, during the vacuum saturation process, the depth of water entering the core is large, and a relatively obvious imbibition effect has occurred, while there are few unactivated pore throats. For pore type II core, increasing pressure can enhance the ability of water entering the core, increase the depth of water entering the core, and a more significant imbibition phenomenon will occur.

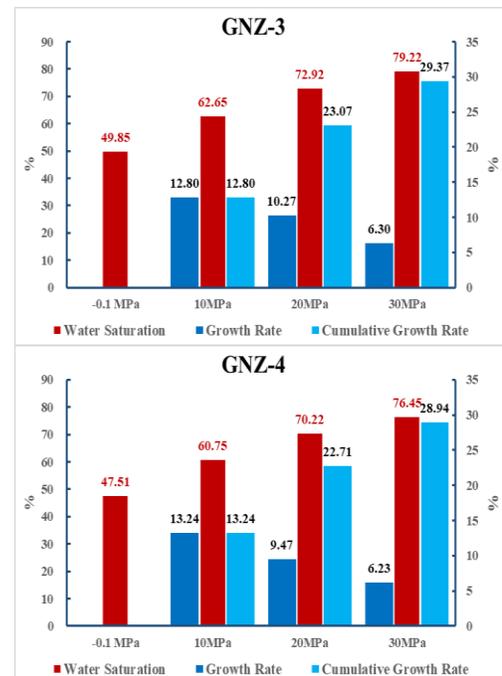


Fig.7. Different saturation pressure dialyzing of type II

3.2.3. Pore type III core analysis

Pore type III cores used in the experiment have permeability of 0.0003mD and 0.0006mD respectively, and porosity of 4.96% and 5.51% respectively. Pore type III cores are the ones with the worst porosity and permeability in this experiment, and there are no cracks in their cores. The main seepage channel is the throat

between the rock particles, but the throat is worse developed than the pore type II core, and its saturation process is significantly different from that of the pore type I and pore type II cores.

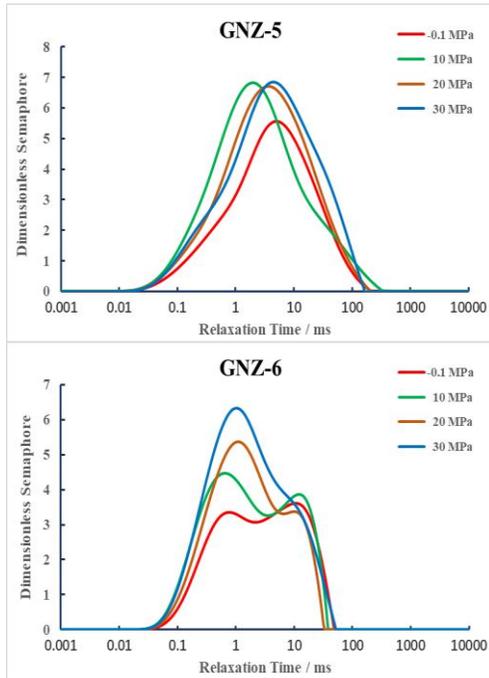


Fig.8 . Different saturation pressures of type III cores

As can be seen from Fig. 8., the main peaks of GNZ-5 and GNZ-6 of pore type III cores are distributed around 1ms under vacuum saturation conditions. When the saturation pressure continues to rise, the T₂ spectrum of pore type III cores mainly moves up to the left side, and the saturation degree of small-size pore throats is greatly affected by the saturation pressure. On the one hand, in pore type III cores, the number of large dissolved pores is small; on the other hand, dense cores have poor seepage capacity and water cannot enter the core. Although the smaller the throat radius, the stronger the capillary effect, water cannot enter the deep part of pore type III cores due to too low permeability, so the imbibition mainly occurs on the core surface and near the large pore throat, and the local imbibition effect is strong. The overall imbibition effect is weak.

As can be seen from Fig. 9., the vacuum saturation of pore type III cores is the lowest, and GNZ-5 and GNZ-6 are 38.56% and 39.21%, respectively. During the vacuum saturation process of pore type III cores, water cannot enter the core depth, and some throats cannot contact water, and no imbibition occurs. With the continuous increase of saturation pressure, water overcomes the resistance and gradually enters the core, and the increases in water saturation of GNZ-5 and GNZ-6

decrease from 6.32% and 5.87% to 3.68% and 3.87%, respectively. The cumulative water saturation increases are 15.01% and 14.41%, respectively, and pore type III cores.

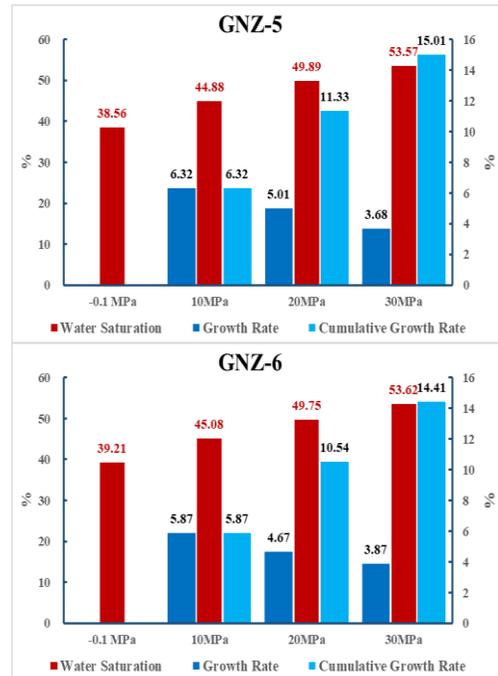


Fig.9. Different saturation pressure dialyzing of type III

4. CONCLUSIONS

(1) The ultra-deep carbonate pore type reservoir is mainly composed of small-size karst caverns, solution pores and intergranular pores, while fractures, half-filled fractures and throats are the main seepage channels.

(2) After vacuum pumping saturated water, the main peak of T₂ spectrum of pore type I core is distributed in 1~10ms, pore type II main peak is distributed in 10~100ms, and pore type III core main peak is distributed in about 1ms.

(3) The existence of micro-fractures can greatly improve the efficiency of core saturated water and the overall imbibition degree of core. The maximum effect of increasing saturation pressure on pore type II core is about 30%, and the minimum effect on pore type III core is about 15%.

(4) The depth of water migration into the core largely determines the overall imbibition saturation degree of the core, and the existence of cracks can increase the overall imbibition swept volume of the core. The local imbibition effect of the core with too low permeability is stronger, but the overall imbibition saturation degree is low because water is difficult to enter the core. The high pressure imbibition efficiency is highest when the permeability is between 0.001 and 0.01mD.

ACKNOWLEDGEMENT

This research is supported by the National Natural Science Foundation of China (51774300).

DECLARATION OF INTEREST STATEMENT

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. All authors read and approved the final manuscript.

REFERENCE

- [1] Aline Machado de Azevedo Novaes,Rafaella Magliano Balbi de Faria,Mônica Antunes Pereira da Silva, et al. Evaluation of rock-fluid interaction in a carbonate reservoir: Backflow test and reactive transport simulations[J]. *Geoenergy Science and Engineering*, 2023, 230: 212158.
- [2] Dianshi Xiao,Shu Jiang,David Thul, et al. Combining rate-controlled porosimetry and NMR to probe full-range pore throat structures and their evolution features in tight sands: A case study in the Songliao Basin, China[J]. *Marine and Petroleum Geology*, 2017, 83: 111-123.
- [3] Hyoung Suk Suh,Dong Hun Kang,Jaewon Jang, et al. Capillary pressure at irregularly shaped pore throats: Implications for water retention characteristics[J]. *Advances in Water Resources*, 2017, 110: 51-58.
- [4] Jingdong Liu,Lei Li,Cunjian Zhang, et al. Identification and quantitative evaluation of pores and throats of a tight sandstone reservoir (Upper Triassic Xujiahe Formation, Sichuan Basin, China)[J]. *Marine and Petroleum Geology*, 2022, 140: 105663.
- [5] Kam Ng,J. Carlos Santamarina. Mechanical and hydraulic properties of carbonate rock: The critical role of porosity[J]. *Journal of Rock Mechanics and Geotechnical Engineering*, 2023, 15(4): 814-825.
- [6] Mohamed Lamine Malki,Mohammad Reza Saberi,Oladoyin Kolawole, et al. Underlying mechanisms and controlling factors of carbonate reservoir characterization from rock physics perspective: A comprehensive review[J]. *Geoenergy Science and Engineering*, 2023, 226: 211793.