Evaluation criteria for gas source rocks of marine carbonate in China*

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Abstract Hydrocarbon generating and expulsion simulation experiments are carried out using samples artifically matched between the acid-dissolved residue of relatively low-maturity limestone and the original sample. This work makes up for the insufficiency of source rock samples with high abundance of organic matters and low maturity in China. The organic carbon content of the 10 prepared samples varies between 0.15% and 0.74%. Pyrolysis data and simulation experiment results of hydrocarbon generating and expulsion, which were obtained by a high-temperature and high-pressure open system, indicate that the lower limit of organic carbon content for marine carbonate rock to generate and expel hydrocarbons is 0.23%—0.31%. In combination with the numerical analysis of organic carbon in marine carbonate rocks from Tarim Basin, Sichuan Basin, Ordos Basin and North China, as well as the contribution of these gas source rocks to the discovered gas pools, we think that the organic carbon criterion for carbonate gas source rocks should be 0.3%.

Keywords: **carbonate gas source rock, gas-generating efficiency, evaluation criteria, simulation experiment.**

Previous researches have shown that the original sedimentary environment, late superimposition, and transformation of basins of the marine sequences in China are different from the typical marine sedimentary basins in other parts of the world[1—4]. In China, a number of large-size oil/gas fields have been discovered in the marine strata of Tarim, Sichuan and Ordos Basin[5—8], making marine strata an important superseding area for hydrocarbons. Therefore, it is very important to probe into petroleum geology, hydrocarbon resource potential, and distribution of marine strata in China.

Marine gas source rocks in China can be classified into two types, that is, clastic rock and carbonate rock. The evaluation criteria for marine clastic rocks are the same as for non-marine mudstone, and this practice has been generally accepted by the Chinese researchers and explorationists. There are many examples demonstrating that high-abundance marine carbonate rocks could, as effective source rocks, form oil/gas pools with great commercial value. Table 1 lists the organic carbon content of some marine gas source rocks in the world.

Table 1. Organic carbon content in typical marine gas source rocks[9]

Basin	Oil/gas field	Gas source rocks	TOC[a)] (%)	Reservoir
East Siberia	Yurubchenskoye	Z_2 mudstone, marl,	0.7—4	Z
		argillaceous dolomite	2.4—8.7[b)]	
Caspian Sea Coastal	Tengiz	C_3—P_1 marl	1.2—4	C
Michigan	Lima-Indiana	O_2 shale & limestone	1.3[c)]	O_2
	Onian-Scypion	O_2 shale & limestone	0.5—1.5	
Williston	Cabin Creek	D_3 shale & marl	3.8	O_2

a) Total organic carbon; b) in depression; c) maxium 4.23.

Concerning about whether the low-abundance (TOC< 0.5%) marine carbonate rocks in China could act as effective source rocks to form oil/gas pools with commercial value, many researches have been conducted but no final conclusion has been reached yet. Taking the genesis of large gas fields in the central Ordos Basin as an example. Huang et al.[10] and Chen[11] believed that the genesis should be of a combined type: both the Lower-Paleozoic marine carbonate and the Upper-Paleozoic continental-

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marine alternating coal strata make contributions, with the contribution proportion at about 50% and 85%—90%, respectively. In accordance with Dai et al.[12,13], the Upper-Paleozoic coal measure strata are the main contributor, while Xia[14] believed that it is mainly contributed by the Upper-Paleozoic coal measure strata, and, even if marine gas source rocks make some contributions, it is contributed by the limestone developed in the Upper-Paleozoic continental and marine alternating strata. Obviously, they have no consistent opinions on such a question. To address this question, we selected acid-dissolved residue of limestone with relatively low maturity and artificially matched it with original samples to prepare samples containing different organic carbon contents. Using these prepared samples, we analyzed rock-eval and established the lower limit value for significant gas generating through hydrocarbon generating and expulsion simulation experiments. In combination with the numerical analysis of organic carbon in gas source rocks from the Chinese marine-genesis gas pools, a reasonable evaluation criterion valuable for explorations of marine carbonate gas source rocks was set up.

1 Lower limit of organic carbon determined by the gas-generating simulation experiment of carbonate rock

1.1 Selection of original samples

The present high-evolution degree of the Chinese carbonate rocks makes themselves very difficult to perform hydrocarbon generating and expulsion simulation experiment. To solve this problem, two approaches have been adopted: the first is to select samples with low maturity and high organic abundance from other countries for the study, e.g. Jordan limestone[15,16]; the second is to use prepared kerogen to conduct simulation experiment under various conditions[16]. However, geological background of these samples is not the same as the Chinese samples, and the geological conditions represented by kerogen data are far from the actual ones. Therefore, we have to prepare a series of samples with distinct abundance of organic matter, avoiding the effects of sedimentary environment and different evolutional degree on experimental data, and making the hydrocarbon generating environment of organic matter (catalytic action of clay minerals and trace elements on gas generating) closer to the actual geological conditions.

Among the Lower-Paleozoic and earlier strata in China, the profile of Majiagou Group, located at Zhaogezhuang, Tangshan in North China, is featured by its low evolutional degree, complete basic field work, and abundant experimental research data. Therefore, samples from this group were selected for this study. After analyzing the basic geochemical data of 27 Cambrian and Ordovician samples, we selected the micritic micaceous limestone of Maer Section of Ordovician System as the original samples (Table 2). The original sample has a low abundance of organic matter (0.13%) and with T_{max} at 435 ℃, apparent vitrinite reflectance at 0.7% (calculated on the basis of asphalt reflectance), hydrogen index at 231 $mg \cdot g^{-1} \cdot TOC^{-1}$, organic matter being Type II, and carbonate content at 80.6%.

Table 2. Original sample and acid-dissolved residue for the simulation experiment

Sample (code)	TOC (%)	T_{max}(℃)	VR_o a)(%)	S_1($mg \cdot g^{-1}$)	S_2($mg \cdot g^{-1}$)	I_H($mg \cdot g^{-1}$)	Type	Carbonate content (%)
Original (y)	0.13	435	0.7	0.02	0.30	231	II	80.6
Residue (S)	1.42	429	0.7	0.33	3.43	242	II	

a) VR_o is marine vitrinite reflectance

1.2 Sample preparation

The original sample was crushed to 60 mesh, then 800 g of the power was mixed with 6 $mol \cdot L^{-1}$ HCl solution, and stirred thoroughly. The residue was washed by distilled water three times and dried in air. After this treatment, the organic matter was relatively enriched, and the organic carbon content increased from 0.13% to 1.42% (Table 2).

At different proportions, the original sample and the acid-dissolved residue were put together to produce 10 samples of organic carbon (Table 3). Pyrolysis analysis was carried out for each sample, and 4 samples above and below the critical condition (based on analysis results) were selected for gas generating and expulsion simulation experiments in a pressure open system, by which their organic carbon content was determined to be 0.17%, 0.23%, 0.31% and 0.51%.

The gas generating and expulsion simulation ex-

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periment was conducted under the pressure of 20 MPa which was created by a hydropress machine. The simulation temperature was from 250 ℃ to 600 ℃ with 8 points in total, and at an interval of 50 ℃. At each temperature point, 80 g prepared sample was heated to the desired temperature at a rate of 50 ℃·h^{-1}, then staying for 48 h, and the resulting gas and liquid were collected quantitatively.

Table 3. Samples of organic carbon prepared

Sample code	27y·1S	22y·1S	16y·1S	13y·1S	10y·1S	7y·1S	5y·1S	3y·1S	2y·1S	1y·1S
Organic carbon (%)	0.15	0.17	0.20	0.20	0.23	0.28	0.31	0.39	0.51	0.74

1.3 Experimental results and discussion

1.3.1 Analysis of pyrolysis data The results of pyrolysis data analysis, the pyrolysed hydrocarbon content of the 10 samples, and the relationship between effective carbon index and organic carbon content are shown in Fig. 1. From Fig. 1, it can be seen that the organic carbon content of 0.23% is a crucial value. For samples with the content above or below this value, their increase rates of pyrolysed hydrocarbons and effective carbon along with the increase of organic carbon are different. The comparison of the fitted curves between Zone I and II shows that when TOC≤0.23%, though contents of pyrolysed hydrocarbons and organic carbons are elevated along with the increase of the content of organic matter, the variation is insignificant; when TOC is higher than 0.23%, pyrolysed hydrocarbons and organic carbons increase substantially along with the increase of the content of organic matter. Therefore, 0.23% is the lower limit for marine carbonate rocks to extensively expel gas.

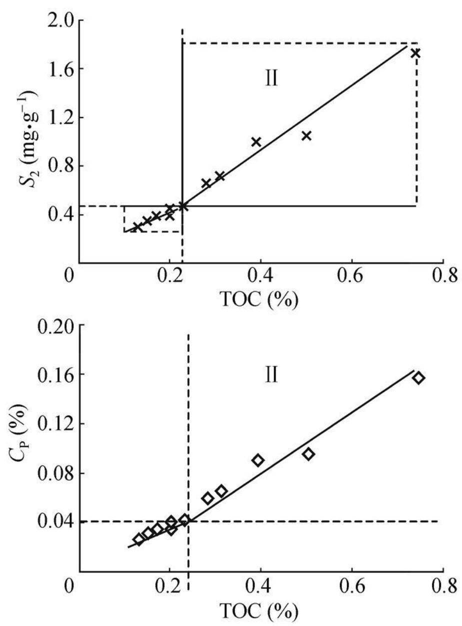

Fig. 1. Variation of pyrolysed hydrocarbons and effective carbon along with the increase of organic carbon.

1.3.2 Gas generating and expulsion experiment performed in the pressure open system In accordance with the increase relations between pyrolysed hydrocarbons and organic carbon, we selected 4 samples for conducting the gas generating and expulsion experiment in a high-temperature and high-pressure open system. Organic carbon contents of the 4 samples are 0.51%, 0.31%, 0.23% and 0.17%, respectively. Fig. 2 shows the by-stage and cumulative gas expulsion volumes of each sample.

The by-stage gas expulsion volume of the 8 temperature points from 250 ℃ to 600 ℃ shows the gas-generating volumes contributed by different organic matter in carbonate rocks at different evolutional stages. According to variations of gas expulsion volumes, the entire process of gas generating and expulsion could be classified into 3 phases:

Phase I: The temperature is below 250 ℃ (Zone I in Fig. 2). During phase I, gas generating and expulsion volume is primarily contributed by soluble organic matter in source rocks.

Phase II: The temperature range is 250—400 ℃ (Zone II in Fig. 2). The distinctive feature of phase II is extremely small gas generating and expulsion volumes. Either the low- or high-abundance gas source rocks have limited or zero expulsion volume, indicating kerogen has not been extensively cracked for generating gas in this phase.

Phase III: The temperature is above 400 ℃ (Zone III in Fig. 2). In this phase, the gas expulsion volume increases largely because of the extensive gas generating by cracked kerogen. However, the increase magnitudes are different for samples with different organic carbon contents. For the two samples with organic carbon content of 0.51% and 0.31% respectively, the by-stage increase rate of expulsion volume is higher, especially at high temperature (≥550 ℃); while for the other two samples with organic carbon content of 0.23% and 0.17% respectively, the increase magnitude is much lower. For the

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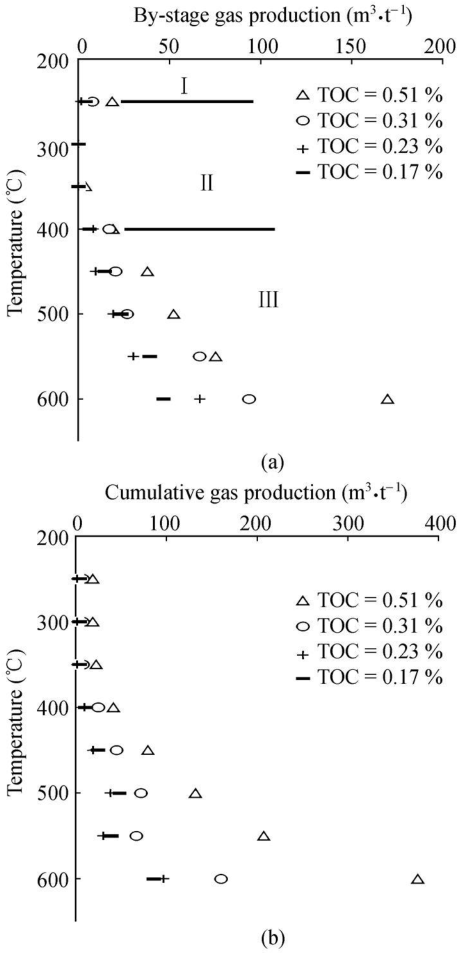

Fig. 2. Comparison of by-stage (a) and cumulative (b) gas expulsion volumes of carbonate samples above and below the critical condition.

latter two samples, their cumulative gas expulsion volumes are almost the same, indicating that, when the organic carbon content is below 0.31%, the total gas generating volume will not increase substantially along with the increase of organic carbon. For the samples with organic carbon content of 0.17% and 0.31%, the increase rates of gas generating and expulsion volume are 0.31% and 0.51%, respectively, reflecting the relation between quantitative and qualitative changes. Therefore, the value 0.31% for the organic carbon content is a lower limit for marine carbonate rocks to generate and expel a large volume of gas.

In addition, a checking and collection device for liquid hydrocarbons was used in the experiment. For the samples with organic carbon content of 0.51%, 0.31%, 0.23% and 0.17%, the cumulative oil generating volumes (sample weight is 80 g) are 12.5, 7.8, 6.8 and 0.1 mg, respectively. Besides the relation with Type II organic matter of source rock, the extremely small oil generating volume is primarily due to that, under the high-temperature condition, the high-energy rate for kerogen to convert directly into gas is predominant. The converting rate is several orders higher than that of the low energy in the initial conversion process from kerogen to oil[17]. Therefore, the direct degradation and gas generating by kerogen mainly occurred at high temperature during experiments.

1.3.3 Theoretical basis for the qualitative change of gas expulsion volume under the critical condition

The essential condition for gas source rocks to expel gas is that the gas generating volume should exceed the gas loss volume, which results from gas adsorption or dissolution by rocks or organic matter, gas diffusion, etc. When the organic carbon content is low, the gas generating volume is small, and the generated gas volume will not exceed the loss volume (i.e. no gas expulsion occurs). Along with the increase of organic matter and when the generated gas volume exceeds the gas loss quantity, gas expulsion occurs. With the increase of the organic carbon content, the accumulated organic matter extensively transforms into effective gas expulsion volume, eventually leading to the qualitative change of gas expulsion volume.

The research on maximal adsorption power of different source rocks indicates that (1) compared with rocks, organic matter occupies the predominant status for gas adsorption power; (2) with the content variation of organic matter, gas adsorption power may vary to several orders; (3) lithology has also certain effects on adsorption power to some extent. Some gas adsorption power data on lithology and different organic matter are listed in Table 4[18].

Table 4. Effect of lithology and content of organic matter on gas adsorption power[18]

Lithology	Organic carbon (%)	Max. gas generating volume ($L \cdot t^{-1}$ rock)	C_{1-5} adsorption power ($L \cdot t^{-1}$ rock)	C_1 adsorption power ($L \cdot t^{-1}$ rock)
Coal	83.25	84193	36000	20828
Calcareous mudstone	23.00	24335	6269	1622
Mudstone	3.73	4711	620	120
Mud-bearing doldsiltite	3.00	2155	518	128

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Considering the experiment results, we think that the gas adsorption by organic matter cannot be interpreted only by the principles of adsorption and super-critical adsorption. So, we put forward the principle of similitude and solutrope to interpret the gas dissolution action by organic matter and oil generated in rocks.

2 Organic carbon value calculated from gas generating

As demonstrated by the Ninth "Five-Year" State Gas Probing Projects and exploring practices, gas generating potential over $20\times 10^8\ m^3 \cdot km^{-2}$ is the main indicator for the forming of large or medium gasfields[7, 19]. In addition, the previous researches have acquired some results on the gas generating and expulsion experiments of carbonate rocks, and lots of simulation experiments of carbonate rocks under various experimental conditions have been undertaken in the past years, and the data of gas generating rates for different types of carbonate rocks from various eras have been accumulated (Table 5). In accordance with the evaluation criteria of gas generating potential ($20\times 10^8\ m^3 \cdot km^{-2}$), organic carbon value could be calculated by the formula of gas generating volume, i.e. the minimal organic matter value for gas generating should exceed $20\times 10^8\ m^3 \cdot km^{-2}$.

The formula of calculating gas generating volume is: $Q_g = C_{org} V \cdot m \cdot K_c \cdot I_g$, where Q_g refers to the yield of gaseous hydrocarbon, C_{org} refers to the content of residue organic carbon, V refers to rock volume, m refers to rock density (for carbonate rock: $2.7\ g \cdot cm^{-3}$), K_c refers to restoration coefficient (1.0 is used), and I_g refers to gas generating rate. Based on the hydrocarbon generating volume per unit, the calculated lower limits of organic carbon are listed in Table 5, varying from 0.23% to 0.48%.

The effective gas expulsion volume equals the result of gas-generating volume minus loss volume. Due to the effective gas expulsion volume could be provided by the experiment, so loss volume is not considered. It is undoubtful that the high-temperature condition in the laboratory has certain expulsion effects on gas expulsion, so the calculated result should be the lowest limit.

Table 5. Gaseous hydrocarbon production rate from simulation experiment and calculated minimal value of organic carbon

Sample	Experimental system	Production rate of gaseous hydrocarbon ($m^3 \cdot t^{-1}$ rock)	Calculated value of organic carbon (%)	Refs.
Limestone of Majiagou, Zhaogezhuang, Tangshan	Enclosed, watering	285	0.31	[20]
Limestone of Taiyuan Group, Hequ, Shanxi		374	0.23	[21]
Limestone of Tieling	Dry combustion of glass tube	178	0.48	[16]
Limestone of Xiamaling		245	0.35	
Limestone of Qinglong		218	0.39	

3 Analysis of organic carbon in carbonate rocks from four main basins in China

Based on carbonate rock horizons of the four main basins in China, the wells with typical characteristics were selected for the analysis of organic carbon. Fig. 3 shows the lithologic profiles and columnar sections of organic carbon content of Tacan 1, Tadong 1 and Tazhong 12 wells located in Tarim Basin; Gaoke 1, Wuke 1 wells and Changjianggou Permian outcrop profile located in Sichuan Basin; Diyu 9, Shancan 1 and Chengchuan 1 wells located in Ordos Basin; and Konggu 3 Well in North China.

The value of organic carbon in the lime-mound facies calcareous-strip marls of Upper Ordovician Series of Tazhong 12 Well in Tarim Basin ranges from 0.26% to 2.17%, with the mean value of 13 samples at 0.75%. As to the under-compensation deep-water basin facies mud-bearing limestone, marl, calcilutite and dolomite sedimented in Tadong 1 Well, the organic carbon content mostly exceeds 2.0%, with the maximal value at 5.52%. As to the Medium-Lower Cambrian evaporation lagoon facies mud-bearing or calcareous dolomicrite and micritic limestone in Tacan 1 Well, the organic carbon content in a few samples is as high as 2.1%, whereas the source rocks with organic carbon content over 0.5% are just 38 m in thickness.

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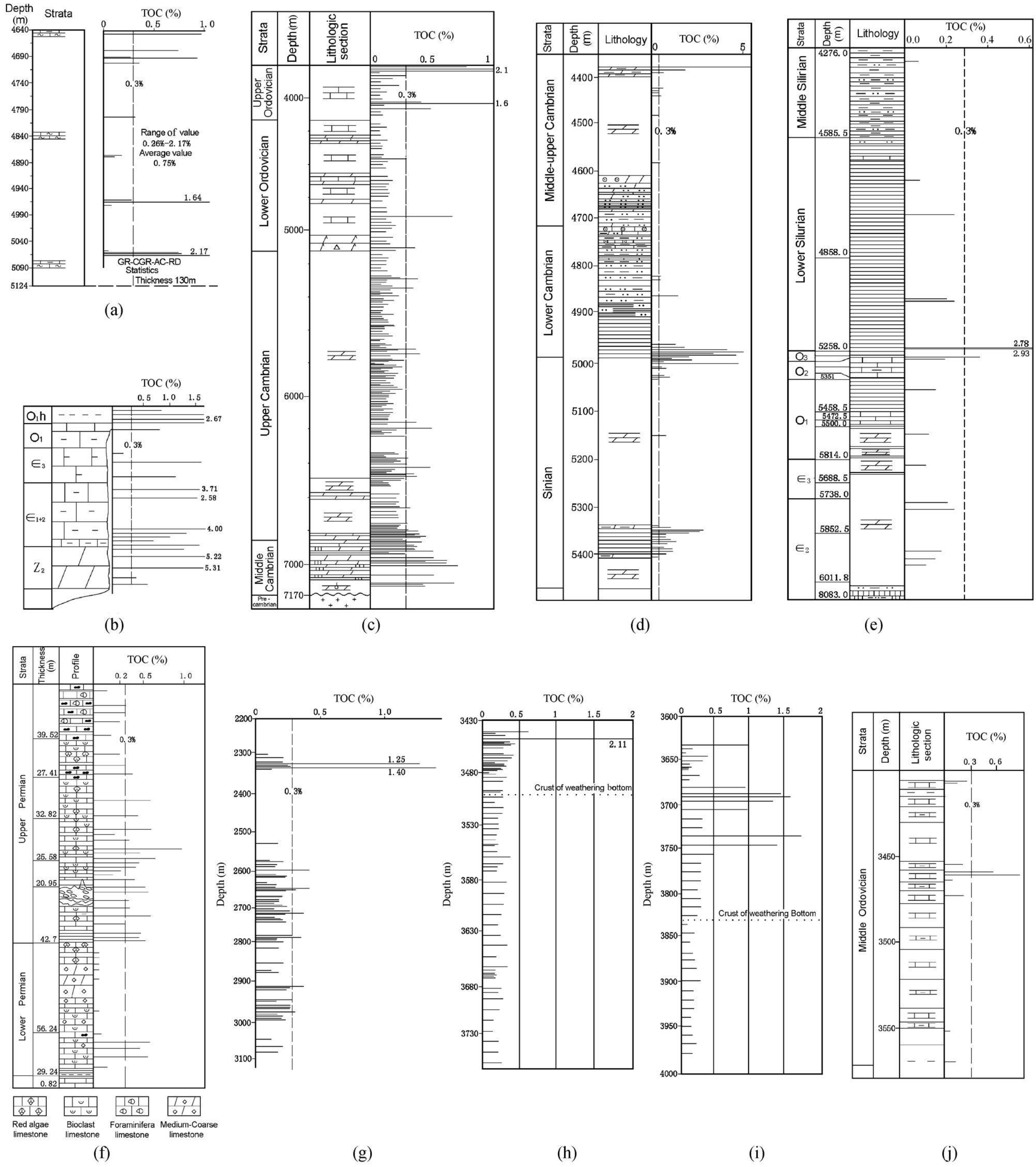

Fig. 3. Profiles of lithology and organic carbon content of the wells in Tarim, Sichuan and Ordos basins, and North China. (a) Upper-Ordovician Series of Tazhong 12 Well; (b) Tadong 1 Well, 4365—4801.3 m; (c) Tacan 1 Well; (d) Gaoke 1 Well, Sichuan Basin; (e) Wuke 1 Well, Sichuan Basin; (f) Changjianggou Permian outcrop profile, Sichuan Basin; (g) Ordovician System of Diyu 9 Well, Ordos Basin; (h) Mawu section, Shancan 1 Well, Ordos Basin; (i) Mawu section, Chengchuan 1 Well, Ordos Basin; (j) Ordovician System of Konggu 3 Well, North China.

With respect to the Lower-Cambrian and Upper-Sinian mudstone and silty mudstone in Gaoke 1 Well, Sichuan Basin, the maximum content of organic matter reaches 5.1%, while that of limestone and dolomite is only 0.10%—1.93%, indicating a quite large change. In Wuke 1 Well, the organic carbon content of Silurian carbonaceous shale is 2.78%—2.93%, whereas a small number of samples from the

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Cambrian dolosiltite and Ordovician limestone have the content of 0.27%—0.46%, and most of them are below 0.2%.

As to the medium-coarse biomicrite in the Changjianggou Permian outcrop profile, the source rock strata with organic carbon content exceeding 0.30% take up about one third of the total thickness of strata.

In Ordos Basin, the organic carbon content is high, for which samples were collected from the Ordovician limestone in Diyu 9 Well, and the limestone weathered-crust zone in Shancan 1 Well and Mawu section of Chengchuan 1 Well. With respect to the carbonate rock below the weathered crust, the content is low, and secondary asphalt predominate in organic matter for the former. With respect to the evaluation of primary hydrocarbon-generating potential below the weathered crust, about a quarter of Ordovician limestone samples have organic carbon content exceeding 0.3%.

The organic carbon content in the Ordovician limestone in Konggu 3 Well in North China is generally low (less than 0.3%), except for a few intervals with higher content due to the infill of secondary asphalt.

Based on the comparison results of 10 profiles, it could be concluded that distribution and hydrocarbon generating performance of marine carbonate source rocks are closely related with the sedimentary environment, which not only controls the lithology and organic matter abundance, but also affects the elemental composition and hydrocarbon generating potential of kerogen. The high-abundance marine source rocks originated in reduction. Weak reduction environment (e.g. under-compensation deepwater basin facies, evaporation lagoon facies, etc.) could form commercial oil/gas pools, such as Weiyuan, Luojiazhai, Wubaiti, Shapingchang, Wolonghe, Dachiganjing, Longmen, Tieshan, Gaofengchang, Xihekou, Fuchengzhai and Shuangjiaba gasfields in Sichuan Basin; as well as Hetianhe, Jilake and Yakela gasfields in Tarim Basin. As to the low-abundance marine strata formed in shallow water — extremely shallow water platform facies—an oxidization-prone environment, no effective gas source rocks would exist unless the organic carbon content reaches 0.3%.

In North China and Ordos Basin, the organic matter abundance of marine strata is generally low. In addition, due to the superimposition and transformation of multi-phase/time tectonic movements, the effective Paleozoic source rocks are distributed sparsely, the evolution and characteristics of source rocks are inconsistent among blocks, and the reservoir forming conditions are complicated. As a result, the exploration difficulty and risk are relatively high.

4 Conclusions

(1) In this research, hydrocarbon generating and expulsion simulation experiment were carried out using the samples artificially matched between the acid-dissolved residue of relatively low-maturity limestone and the original samples, which makes up for the insufficiency of source rock samples with high abundance of organic matters and low maturity in China. Organic carbon content of the 10 prepared samples varies from 0.15% to 0.74%. In accordance with the pyrolysis data as well as the hydrocarbon generating and expulsion simulation experiment results of high-temperature and high-pressure open system, the lower limit of organic carbon content for marine carbonate rock to significantly generate and expel hydrocarbons should be 0.23%—0.31%. Moreover, in combination with the numerical analysis of organic carbon in marine carbonate rocks from Tarim, Sichuan, Ordos Basin and North China, 0.3% could be regarded as the evaluation criterion of the lower limit of organic carbon content for carbonate gas source rocks. With respect to the carbonate rock with organic carbon content less than 0.3%, the gas generating volume is limited and almost totally lost, belonging to ineffective gas-generating volume. While for carbonate gas source rock with organic carbon content higher than 0.3%, the gas generating volume exceeds the loss quantity and reaches the expulsion threshold, and the gas expulsion volume improves significantly along with the increase of organic matters.

(2) The purpose of taking 0.3% (lower limit of organic carbon content) as the evaluation index of marine gas source rocks is to facilitate its application in gas exploring activities. However, the quantitative evaluation of gas generating potential of carbonate source rocks is dependent on the organic facies zone formed in the sedimentation of gas source rocks, oxidization-reduction conditions during the sedimentary-digenetic process, and the thermal evolution degree, etc. Organic facies of restrained marine sedimentation

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is the most favorable environment for the development of carbonate gas source rocks[1)]. The humification during sedimentary - diagenetic process may deteriorate the organic matter type in carbonate gas source rock[20]. While for highly-evolutional carbonate gas source rocks, the residue organic carbon content and hydrocarbon generating potential are reduced due to the generation of large-quantity hydrocarbons, but the original organic carbon content was higher than the present one[20].

(3) The evaluation of marine carbonate gas source rocks is not only related with the original gas generating potential, but also closely related with the gas generating process and reservoir forming process. During the hydrocarbon generating process of carbonate rocks, the oil-gas ratio is high, and the hydrocarbon generating history is quite complicated at the bottom of sedimentary superimposition basins. This will result in the secondary hydrocarbon generation process, the forming of paleo-reservoirs, and the gas generation by oil cracking. The forming of paleo-reservoirs is an enrichment process for organic matter in marine carbonate source rocks, which makes possible the turning of carbonate source rocks into high-quality hydrocarbon resources. Moreover, the dual attributes of carbonate rock make reservoir carbonate rock turn into a special kind of gas source rocks due to the infill of secondary asphalt.

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