Evaporation Stage of Paleo-saline Lake in the Sichuan Basin, China: Insight from Fluid inclusions in Halite

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Abstract: Sichuan Basin is one of the most important marine–salt forming basins in China. The Simian and Triassic have a large number of evaporites. The Triassic strata have found a large amount of polyhalite and potassium-rich brine. However, no soluble potassium salt deposit were found. In this study, the halite in well Changping 3 which is located at the eastern part of the Sichuan basin was studied using the characteristics, hydrogen and oxygen isotopes of the fluid inclusion in halite to reconstruct the paleoenvironment. The salt rocks in well Changping 3 can be divided into two types: grey salt rock and orange salt rock. The result shows that the isotopic composition of the halite fluid inclusion is distinct from the global precipitation line reflecting that the salt formation process is under strong evaporation conditions and the climate is extremely dry. At the same time, compared with the hydrogen and oxygen isotopes of brine in the Sichuan Basin and the hydrous isotope composition of the inclusions in the salt inclusions of other areas in China, it is shown that the evaporation depth of the ancient seawater in the Sichuan Basin was high and reached the precipitation of potassium and magnesium stage.

Key words: hydrogen and oxygen isotopes, fluid inclusions, halite, Triassic, Sichuan Basin

Citation: Ding et al., 2020. Evaporation Stage of Paleo-saline Lake in the Sichuan Basin, China: Insight from Fluid inclusions in Halite. Acta Geologica Sinica (English Edition), 94(3): 801–808. DOI: 10.1111/1755-6724.14398

1 Introduction

Halite and potash deposits can be easily dissolved and are very difficult to be preserved. After precipitation, subseguent reworking and destruction (such as digenesis, tectonic activities) often result in great loss in geological information obtained from the original salt deposit formations, thus preventing correct insight into the sedimentary environment for the salt deposits and their salt-genetical mechanism. Abundant original geological information is saved in salt mineral fluid inclusions called living fossil preserved in evaporates, which can provide direct, quantitative information for study of the ancient temperature, composition of ancient waters, and compositions of the ancient atmosphere (Roberts et al., 1995).

Thanks to the increasing advance in techniques for testing of inclusions in recent years, analyses of the salt inclusions with respect to their compositions and temperature have been widely applied in researches revealing the sources of the salt-genetic substances, and inversing their sedimentary environments (Roedder et al., 1984; Roberts et al., 1995; Lowenstein et al., 1998; Bension et al., 1999; Liu et al., 2005; Meng et al., 2011, 2013), thus undoubtedly providing a good chance for insight into the setting for salt deposition and sedimentary patterns at the geological ages. The hydrogen and oxygen isotope were tested by high temperature burust method (Ding, 1986; Zheng et al., 2000).

Hydrogen and oxygen isotopes are often used as parameters to distinguish whether the formation waters come from the atmospheric precipitation or from ocean (Luders et al., 2010). The remnant lake water is often trapped in evaporate minerals, forming fluid inclusions, which can be used to evaluate the characteristics and evolution of the chemical compositions. Halite is one of such minerals. Hydrogen and oxygen isotopic compositions in halite inclusions can be used for identification of fluid source, as well as processes for their mixing and evolution (Knauth et al., 1986; Horita et al.,1990; Goldstein, 2001; Thomas et al., 2011; Lowenstein et al., 2011). Additionally, laser Raman of halite inclusions can be used to inverse compositions of ancient lake water (Sun et al., 2013). In this paper, Authors intend to analyze fluid inclusions in halite rocks from newly drilled cores of the Changshou area in the east part of the Sichuan basin, regarding their characteristics, hydrogen and oxygen isotopic compositions and laser

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ablation, so as to restruct the ancient environment of the basin where halite and potash rocks are generated, and to conduct a preliminary discussion on the methods for supply of the salt-genetic materials.

2 Geological Settings

The Sichuan Basin is a rhombic structural-sedimentary basin in the upper Yangtze Para-platform that was formed by intersection of the NE–SW-trending and NW–SE-trending deep faults (Zhai, 1989; Lin et al., 2007; Meng, 2011). The basin is surrounded by a series of fold and thrust belts (Fig. 1). The western portion of the basin is bounded by structural elements produced by the collision of the eastern Tibetan Plateau with the Yangtze Block. To the southwest, it is bounded by a left-lateral fault, which is part of the Songpan–Ganzi fold belt. To the northwest, the basin is bounded by the Longmenshan thrust belt. To the north, the basin is bounded by a thrust belt part of the Qinling orogenic belt, which was formed by the collision of the Tibetan Plateau, North China Block, and Yangtze Block. To the northwest, it is bounded by the Dabashan thrust belt, which was produced by the collision of the Yangtze Block and the North China Block. To the southeast and the south, the basin is bounded by structural elements that were produced by the collision of the South China Block with the Yangtze Block. The Qianyuxiang fold belt bounds the basin to the southeast and the Micangshan dome bounds the basin to the south (Fig. 1). In the early Triassic, the Sichuan Basin was located on a passive continental margin at the eastern edge of the Paleo -Tethys Ocean (Mao et al., 2006). An abundant seawater supply and evaporation resulted in the deposition of large amounts of salts (Lin et al., 2003; Zhang et al.,2018). During the late Triassic, the Sichuan Basin was transformed from carbonate-evaporitic sediments to fluvial-deltaic sediments and from cationic basin to foreland basin (Liu et al., 2006).

In the Early Triassic, sea-level rose all the world (Haq et al, 1987; Miller et al., 2005), while the Yangtze plate was raised due to the intracontinental collision of the Yangtze and North China, and the sea-lever declined at the same time, finally formed Latin period all the Yangtze plate (Yin, 1982; Cheng et al., 1994; Zhao et al., 1996; Mei et al., 1997; Yang et al., 2000). In the early Triassic (Jialingjiang Formation) the sedimentary environment was mostly coastal shore of Sabah (Fig. 2), Sabah platform, Shore plateform. The time of the T_1j^3–T_1j^4, is an important evaporite sedimentary period in this area, and the coast was expanded from west to east for a transgressive-retrogressive cycle. The sedimentary environment was transformed from sediments of the lagoon and tidal flat to Sabah and salt lake sediments. the last period of Jialingjiang Formation, the crustal uplift, the Yantze platform uplift out of the sea, the Jian'an ancient land was formed at the east part of the Yangtze platform, at the same time, the volcano erupted, all the platform and surrounding were deposited about 1 meter thick volcanic sediments (Guan et al., 1990).

The outcrop of the salt-bearing stratum in the Sichuan Basin is middle-lower Triassic in age. The lower part of

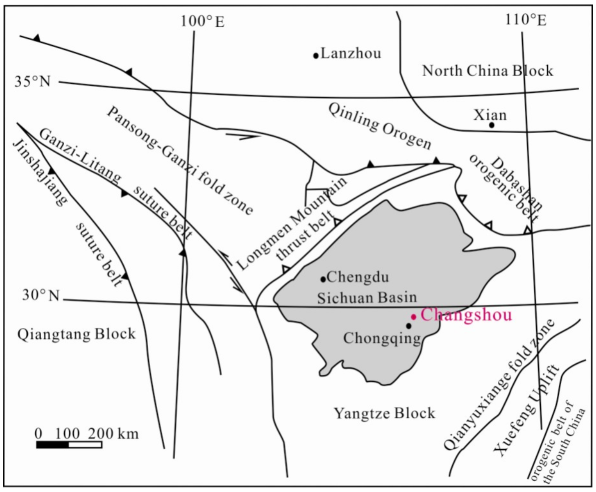

Fig. 1. Map showing the location of Sichuan Basin (modified from Meng, 2011).

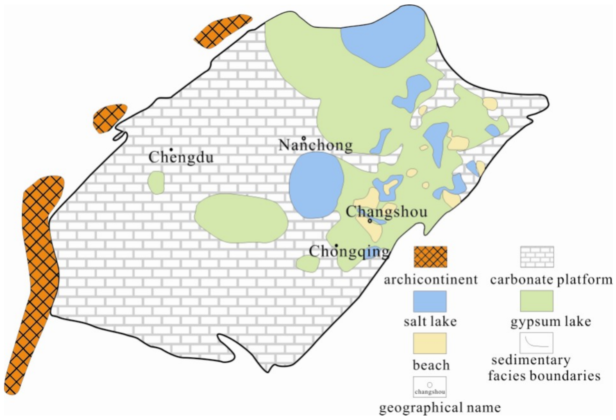

Fig. 2. Lithofacies and paleogeography sketch of the fourth member of Jialingjiang Formation, Sichuan Basin (modified from Meng, 2011).

the lower Triassic is composed of the Feixianguan Formation (T_1f), and the upper part is Jialingjiang Formation (T_1j). During the early Triassic, the entire Sichuan Basin underwent transgression, and the Feixianguan Formation (T_1f) was composed of purple shale with marlite, limestone and oolitic limestone, which belonged to the shore shallow sea facies that was not salt-bearing. The Jialingjiang Formation (T_1j) can be divided into five members: the first member (T_1j^1) is a large suite of gray-dark to gray micrite limestone with some marmite and gypsum; the second member (T_1j^2) is dominated by gray dolomite, anhydrite, and rock salt with anhydrite, cream dolomite and muddy dolomite; carbonates are the main rock types in the third part (T_1j^3), and there are limestones with some dolomitic limestone; the fourth and fifth sections (T_1j^4, T_1j^5) are dominated by thick anhydrite, rock salt and cream dolomite (Xu et al., 2012a, b). Two of the members (T_1j^1 and T_1j^3) and two submembers of T_1j^5 (T_1j^{5-1} and T_1j^{5-2}) were deposited during transgressions. The remaining members (T_1j^2 and T_1j^4) and submember of T_1j^5(T_1j^{5-3}) were formed during highstands (Hu et al., 2010). During deposition of the high stand systems tract, restricted lagoons and tidal flats were developed during high-frequency, short-term regressions (Hu et al., 2010).

The study region of this work, the Changshou area, is in the fold belt of the eastern Sichuan Basin. During deposition of the fourth member of the Jialingjiang Formation, many small restricted sags with shallow water were developed around the Dianjiang–Liangping–Wanxian gypsum basin, with the Dianjiang serving as the depocenter for evaporites (Meng, 2011) (Fig. 1). Many organic banks were developed in the Dianjiang–Liangping area (Meng, 2011; Xu et al., 2012), indicating the gradual evolution of the sedimentary environment of the basin from a restricted sea to lagoon (Lin, 2003) and subsequently closed. Around Well Changping 3 (Fig. 3), the second submember in the fourth member of the Jialingjiang Formation developed three layers of rock salt consisting of anhydrite and dolomite.

The top of this drilling is dominated by the gray anhydrite with multiply layer of dolomite with a thickness of ten centimeters to several tens of centimeters. The middle layer is composite of the anhydrite-bearing halite and anhydrite. The bottom part is about the grey anhydrite

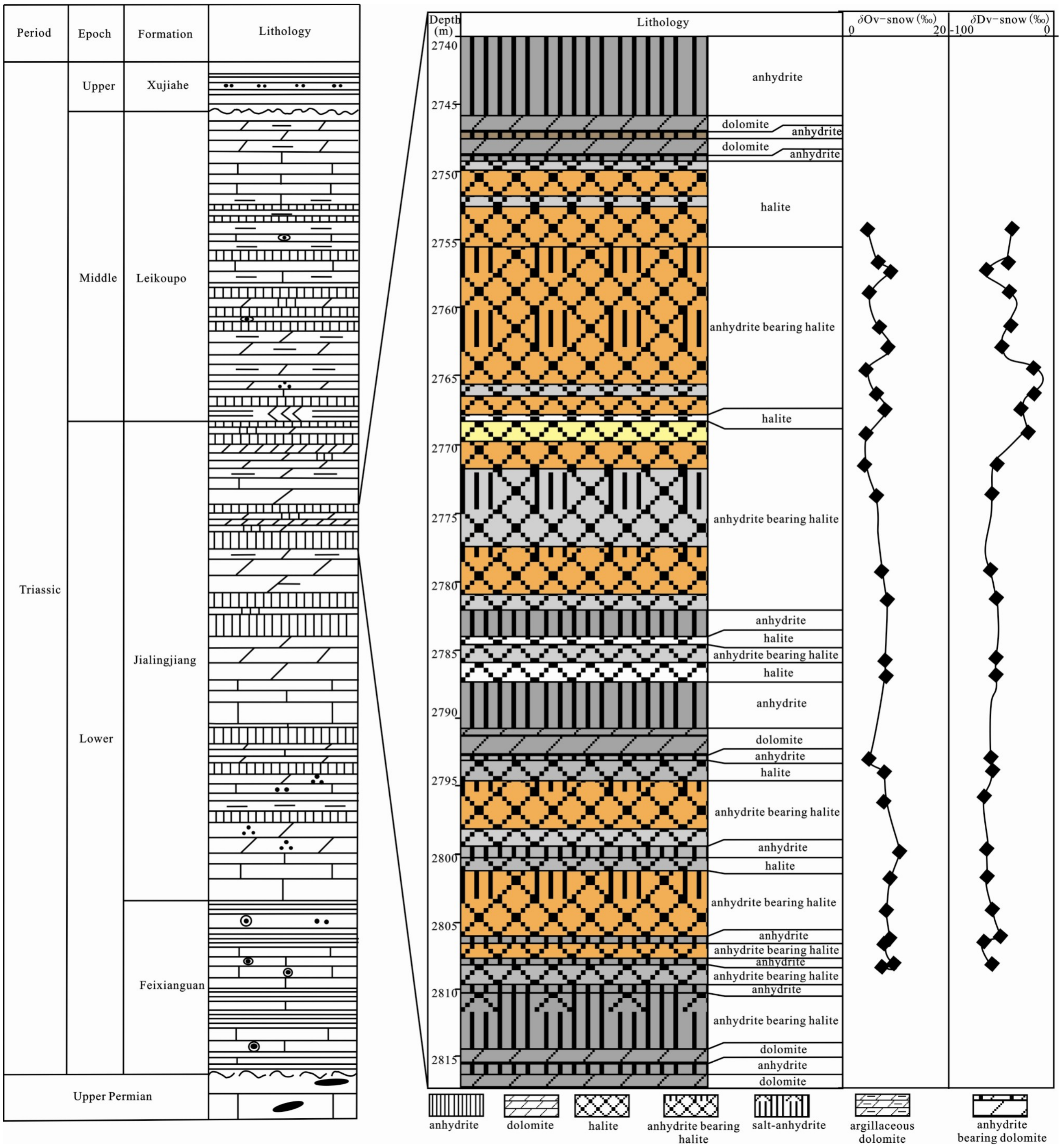

Fig. 3. Composite stratigraphy illustrating the rhythmic nature of the Triassic formation and the fourth member of Jialingjiang Formation of Well Changping3, Changshou (modified by Chen, 2010).

and dolomite. From the top to bottom, the formation tell the story about the life of the salt lake: the bottom part is the sulfate (carbonate), which means the born of the salt lake, the halite tells the time for the salt lake developed, the top part ‘s sulfate (carbonate)

3 Characteristics of Evaporite Formation

The second submember of the fourth member (T_1j^{4-2}) of the Jialingjiang Formation in the Changshou area, Sichuan Basin, is characterized by anhydrite interlayered with thin layers of dolomite and salt-bearing series. In this study, a core was obtained from Well Changping 3 in Shuanglong, Changshou. This area was the term lake in the Dianjiang area; it was surrounded by large gypsum lakes during the deposition of the Triassic Jialingjiang Formation (Fig. 2). Sampling was conducted in the depth interval of 2747.77–2809.00 m, with a total length of 61.23 m. Samples were collected at intervals of 10 to 25 cm in 2012, and all the samples were kept in drier.

In Well Changping 3, the top of the core (T_1j^{4-2}) is mainly composed layers of dark to light grayand brown anhydrite interlayered with dolomite. The middle part of the core (2749.26–2809.00 m) is rock salt with interlayered thin anhydrite layers. The rock salt is orange, light gray or colorless, medium to fine grained, hypautomorphic-allotriomorphic granular crystal (Fig. 4). In the salt-bearing series, anhydrite occurs as both strips and irregular nodules. The width of the strip is 0.5–15 cm. In the upper part (2749.26–2780.82 m), halite around anhydrite nodules has maroon color and generally consist of small crystals (Fig. 4a–c). In the lower part (2780.82–2809.00 m), the majority of the halite in the section is gray to colorless and transparent (even if in contact with anhydrite nodules) (Fig. 3d–e) with relatively larger crystals. At the bottom of the salt-bearing series, salt-anhydrite rock and bedded anhydrite are interlayered with grayish brown to light gray dolomites.

4 Sampling and Methods

In order to prevent the deterioration in accuracy of the test results from mixture with other minerals in the halite rocks, 45 typical halite samples were analyzed for mineral compositions with powder crystal diffraction before this inclusion test. The diffraction was conducted using D/max -rA12kw X-ray diffractometer made by Rigaku Corporation in Japan, with results listed in Table 1. The results indicate that the halite rocks generally have high salt content, except that some halite rock samples contain trace or few gypsum and anhydrite minerals. Through the semi-quantitative analysis of the mineral compositions, those pure halite rocks with high halite content were chosen for test analysis.

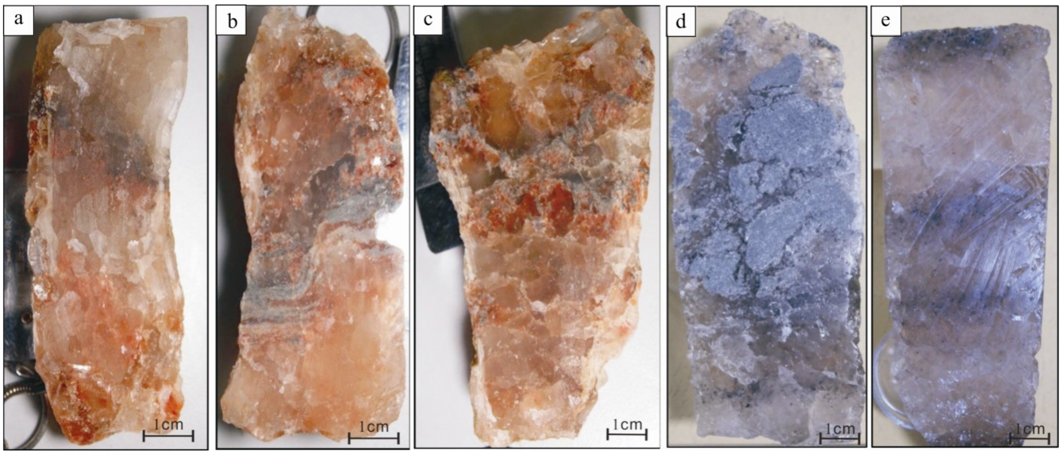

Fig. 4. Typical photos of cores from the salt-bearing series of the fourth member of Jialingjiang Formation from the borehole of Well Changping3, Shuanglong, Changshou.

Table 1 Hydrogen-Oxygen composition of halite fluid inclusions in the fifth member of the Jialingjiang Formation in Core CP3, East of the Sichuan basin, China

Sampling	Deapth(m)	$\delta D_{v\text{-}snow}$(‰)	$\delta O_{v\text{-}snow}$(‰)	Color	Sampling	Deapth(m)	$\delta D_{v\text{-}snow}$(‰)	$\delta O_{v\text{-}snow}$(‰)	Color
LG16	2750.47	−74.1	7.7	orange	LG191	2779.43	−84.4	8.3	grey
LG25	2752.09	−71.2	8.2	orange	LG201	2781.62	−79.1	9.6	colorless
LG39	2754.36	−66.0	5.6	orange	LG224	2786	−81.0	9.2	grey
LG51	2756.74	−69.0	7.7	orange	LG230	2787.09	−80.7	9.3	grey
LG57	2757.36	−84.1	10	orange	LG243	2793.3	−85.2	5.9	grey
LG67	2758.92	−68.4	5.8	orange	LG248	2794.2	−82.3	9.0	grey
LG79	2761.49	−66.3	7.7	colorless	LG259	2796.34	−90.0	8.9	colorless
LG86	2763	−76.2	9.5	grey	LG282	2800.13	−85.9	12.3	grey
LG96	2764.67	−44.6	5	orange	LG301	2802.14	−87.7	10.1	grey
LG108	2766.57	−41.8	7.3	orange	LG308	2804.44	−83.0	9.3	colorless
LG116	2767.63	−57.0	9	orange	LG313	2806.48	−78.4	10.2	colorless
LG128	2769.48	−48.9	5.2	grey	LG315	2806.88	−87.6	9.1	colorless
LG142	2771.64	−77.3	4.8	colorless	LG325	2808.44	−83.8	10.6	colorless
LG156	2773.97	−80.6	7.3	grey	LG328	2808.59	−83.4	8.7	grey

Hydrogen and oxygen isotopes were performed on inclusions of single liquid phase within halite crystals. During the analyses, halite samples with well-preserved fluid inclusions were chosen. Then, halite crystals were bathed and cleaned with pure ethyl alcohol. After cleaning, halite was solarized, powdered and seized with 40–120 mesh, and then was stored in a dryer before analyses. The measurements were done with the Finnigam MAT 235 EM mass spectrometry at the Analysis and Test Research Center of the Beijing Geological Institute of the Nuclear Industry Department and the Isotope Sector of the Mineral Resources Institute of the Chinese Academy of Geological Sciences. Hydrogen isotopes were analyzed through taking water using explosive approach, and preparing oxygen with the zinc approach, at the explosion temperature of 550°C. The hydrogen isotope analysis results with SMOW mean reaching the standard, with accuracies over 2‰. The oxygen isotope studies were conducted in a 10–3 Pa vacuum condition. The water in the inclusions containing no oxygen's minerals was reacted with BrF_3 at a constant temperature of 300°C for 20 minutes, generating O_2, which was cleaned through freezing, and using platinum as catalyst. Then, O_2 reacted with graphite at a constant temperature of 700°C, yielding CO_2. Next, oxygen isotopic compositions were obtained from the samples with the MAT253 gas isotope mass spectrometer. The oxygen test results with SMOW mean reaching the standard, with accuracies exceeding 2‰.

5 Result

5.1 The type of the fluid inclusion

The halite fluid inclusion was developed in the red and grey halite. Previous research indicated that the primary fluid inclusions in the halite are composed of parallel inclusions stripes in the funnel-shaped crystals on the surface of the water body and the lab did crystal inclusion stripes formed at the water bottom. The $\delta^{18}O$ and δD values of water in the primary halite inclusions in simple liquid phase reflect the isotopic compositions during each salt crystallization stage in the ancient brine water lake, which can be considered basis for prediction of the brine evolution. A variety of fluid inclusions occur in the halite rocks of the Early Triassic Jialingjiang Formation in the well Changping 3, this phenomenon can be regard as primary inclusions, which are characterized by simple liquid phase, parallel arrangement, and obliquely intersecting cleavage planes (Fig. 5).

5.2 The hydrogen and oxygen isotopes of the fluid inclusion

The data results show that the $D_{v\text{-snow}}$ values of the inclusion are generally negative. The former values range from the minimum −90 ‰ to the maximum −41.2‰, averaging −74.9‰; and for the $\delta O_{v\text{-snow}}$, 4.8‰ to 12.3‰, averaging 8.3‰. As shown in the figures, the measurement values of the hydrogen and oxygen isotopes are all below the meteoric water line, indicative of a strong evaporation environment and setting.

6 Discussion

From the Figure 6, the values show that the hydrogen and oxygen isotopes are all below the meteoric water line, indicative of a strong evaporation environment and setting at the deposition time. Wang et al. (2015) tested the

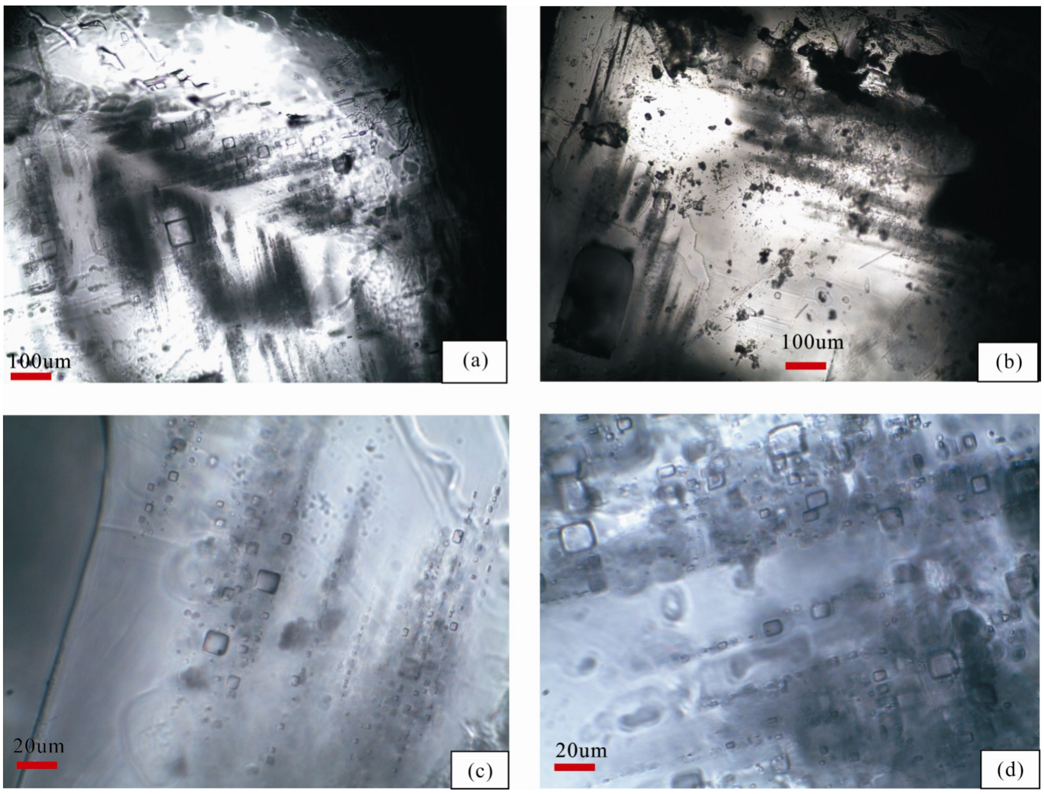

Fig. 5. The picture of the fluid inclusion.

homogenization temperature of the fluid inclusion, with the highest temperature about 63.5°C, and the lowest temperature of about 17.7°C, the average temperature is about 33.7° C. From the isotopes data and the homogenization temperature, it can know that temperature is extremely high, the type of the paloclimate is about the dry and high temperature.

In this study, the hydrogen and oxygen isotopes didn't change frequency. With the decreasing depth, the tendency for the hydrogen isotopes became smaller and the oxygen isotope had a different tendency. It can be seen

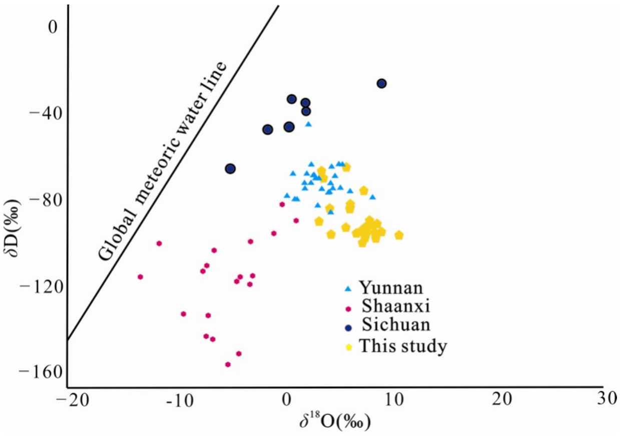

Fig. 6. The hydrogen and oxygen isotopes for the fluid inclusion in halite (Data from Lin et al., 2010; Shen et al., 2017; Ding, 2018).

that the isotopes of hydrogen and oxygen in the deep part are infrequently changed, while in the shallow part, the changes are more frequent. The most drastic changes in the data are in the orange-salt-rock salt part. In the orange-salt-salt section, the lowest value is −84.1‰, the highest value is −48.9‰, and in the non-orange stone salt, the highest value is −90‰. The maximum value is −78.3‰. The orange anhydrite bearing halite contains a small amount of polyhalite, the polyhalite occurs as a petal near clumped aggreates, in which the polyhalite is granular and is distributed among the halite crystals. Polyhalite aggregates usually contain anhydrite within themselves (Fig. 7a–b) or on their edges (Fig. 7c–d), suggesting that the polyhalite was formed from the metasomatism of anhydrite. Halite on the periphery of polyhalite aggregates is usually clearly maroon in color, indicating that the salt lake has a high degree of evaporation and enrichment, but is affected by the relatively light incoming water rich in Ca^{+2} and $SO_4^{-2,}$ and the sedimentary environment is unstable.

Lin et al. (2001) conducted an oxygen and oxygen isotope test on the brine in the Sichuan Basin and used four types of brine moisture (Fig. 6). Triassic brines are mainly marine sedimentary type and marine deposition-atmospheric precipitation superposition type. By comparison, it can be seen that the hydrogen and oxygen isotope values of the Triassic lower fluid inclusions tested in this study have the same interval and consistent distribution trend as that of the Triassic lower-middle system. At the same time, Lin et al. (2001, 2003) believed that the Lower Triassic brine was the residual seawater in

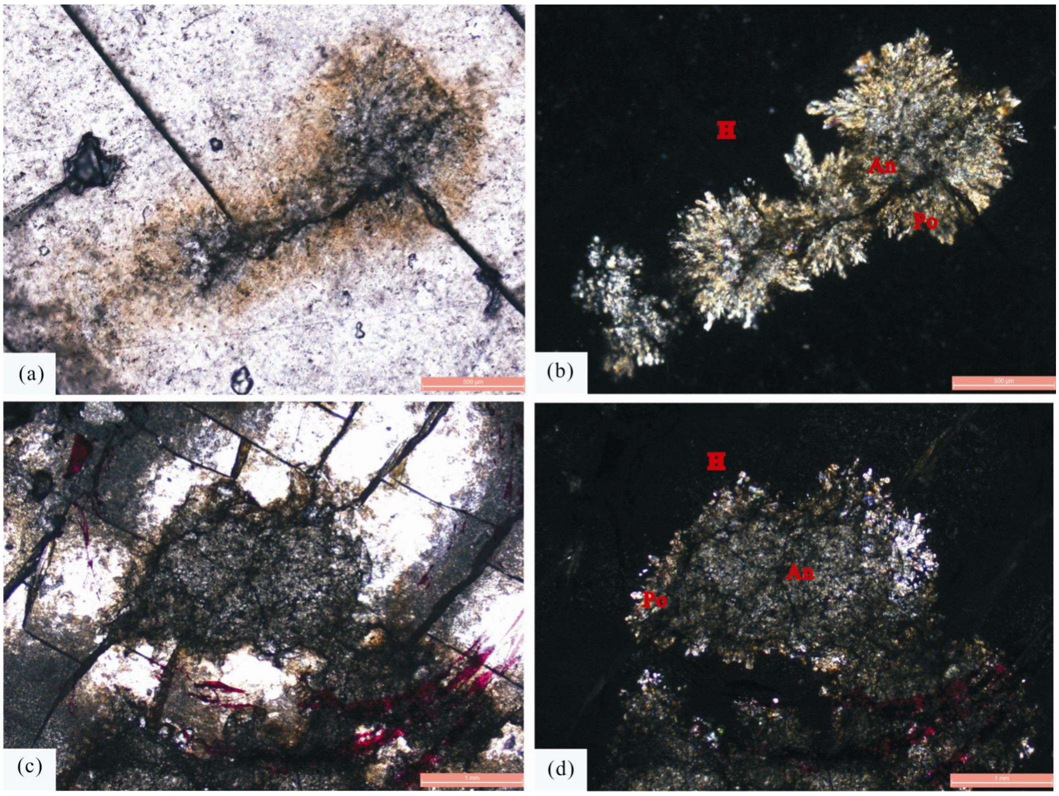

Fig.7. Microscopic characteristics of polyhalite.

(a) and (b) petaline polyhalites developed among halite crystals and centering on anhydrites. Single polyhalite is granular or columnar and halite on the edge are obviously red in color; (c) and (d) massive anhydrites among halite crystals with the edge characterized by metasomatism by polyhalite.

the closed basin, and its cause is closely related to seawater. Therefore, by comparison, it can be concluded that the source of water in the fluid inclusions is same as the brine in the Lower Triassic.

Figure 6 shows the hydrogen and oxygen isotopes of the fluid inclusions of the halite in the areas covered by potassium deposition in China. The values of hydrogen and oxygen isotopes measured by the Institute are roughly in line with the data of Yunnan and Shaanxi (Shen, 2016; Ding, 2018). In the Late Cretaceous Mengyejing Formation in Yunnan, there was the deposition of potash salt, and in Shaanxi, there were also reports of the deposition of potassium rock. This shows that Sichuan also reached the stage of precipitation of potassium and magnesium salt during the Triassic, and the seawater was highly condensed. The paleo-climate at the time of formation was hot and dry.

The hydrogen and oxygen isotope composition of seawater is relatively stable. However, under evaporation, there is a lack of freshwater supply. δD and δ^{18}O are positively correlated with salinity in a certain range, but when they come to concentrated brines, the isotope composition and salinity are negative correlation. As can be seen in Figure 6, the salinity of the brine reflected by the hydrogen and oxygen isotopes of this study has reached 400 g/l (Fig. 8). This is consistent with the composition of inclusions measured by laser ablation method by Sun et al. (2016). This may indicate that in the Triassic, the evaporative enrichment of the brine in the Sichuan Basin exceeds the concentration of the current salt lake, which indirectly shows that the Sichuan Basin has reached the stage of precipitation of potassium and magnesium in the Triassic.

7 Conclusion

(1) In the eastern part of the Sichuan Basin, the primary inclusions of the salt in the fourth section of the Jialingjiang Formation developed, suggesting that the salt deposition environment is a shallow-water depositional environment; the changes in the data of the salt inclusions in the study over time indicate that the sedimentary environment was not present at the time. Stability and transgression was more frequent.

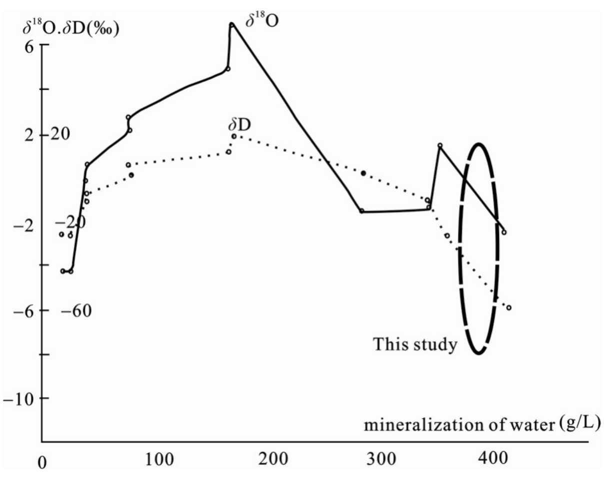

Fig. 8. The relationship between the hydrogen, oxygen isotopes and the salinity (modified from Li et al., 1995).

(2) The hydrogen and oxygen isotope composition of the fluid inclusions in the rock salt obviously deviates from the global atmospheric precipitation line and is in a strong evaporation region. This shows that in the period of the fourth stage of the Jialing River, the climate in eastern Sichuan was arid and the seawater was highly concentrated.

Acknowledgements

This study was supported by the National Natural Science Foundation of China (Grant No.41902065, 41772197), Open Reserch Fund from the State Key Laboratory Breeding base of Nuclear Resources and environment (Grant NRE1803), the Chongqing Science and Technology Innovation Project (Zhu Zhengjie), the Major State Basic Research Development Program (No.2011CB40300). Professor Jun Tang, Yongfei Zheng from the University of Science and Technology of China, Professor Randy Stoler from the University of Kansas gave a great help in manuscript writing process. Professor Tim. K. Lowenstein from Binghamton University and Robert Goldstein from Kansa University give some advices on finding the fluid inclusion types.

Manuscript received Nov. 1, 2018
accepted Apr. 9, 2019
associate EIC: CUI Peng
edited by FEI Hongcai

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