

PS 4D Time-Lapse Characterization of Porosity Evolution in Organic-Rich Shales*

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Abstract

The porosity evolution in shales is more complex than that in sandstones, due to special hydrocarbon (HC) generation in shales. Lots of scientists are trying to interpret this process via SEM observation on samples with different maturities, which is view-based and can provide useful information. However, it is too difficult to find the same organic-matter (OM). Artificial-maturation is a good way to get relatively homogeneous samples, and can provide useful in-situ information to understand changes at different maturity. The current studies only use gas adsorption to provide quantitative analysis on whole pore system. However, they cannot separate OM pores from inorganic pores. Therefore, a new method that can provide information on different pores is urgently needed to help to understand the porosity changes in shales. In this study, we use the physical modeling under high temperature and pressure and X-ray nano-CT scanning data to analyze 4D porosity evolution with increasing maturity. Other data including nitrogen adsorption, XRD, TOC, Rock-eval, and SEM observation on the same position before and after physical modeling help to find the in-situ changes with increasing maturity. The pressure in physical modeling is 80 MPa and the temperature includes 300°C, 350°C, 450°C, and 550°C. The sample is the low mature Chang 7 Shale with Type II kerogen from the Ordos Basin. TOC = 2.23%, Ro = 0.67%. S1 = 5.1 mg/g, S2 = 10.73 mg/g. The preliminary results are as follows:

- (1) Porosity evolution is positively related to maturity according to the porosity data at different evolution stages. More and more nano-pores are developed in organic-rich shale with the maturity increasing. The porosity which is calculated from nano-CT scanning model increased from 0.56% to 2.06% when temperature increased from 20°C to 550°C.
- (2) The process of porosity evolution can be divided into two parts. First, porosity increases rapidly when the maturity increasing from low mature stage to oil window and gas window and abundant organic matter cracks to generate HC. Then, porosity system keeps stable when the shale enters into over-mature stage and the intensity of both HC generation and compaction decreases.
- (3) The percentage of new-created porosity in shales varies among organic matter evolution, clay mineral transformation, and brittle mineral dissolution, and the ratio is 6:3:1 respectively. Abundant organic matter pores occurs when the maturity over 1.2%.

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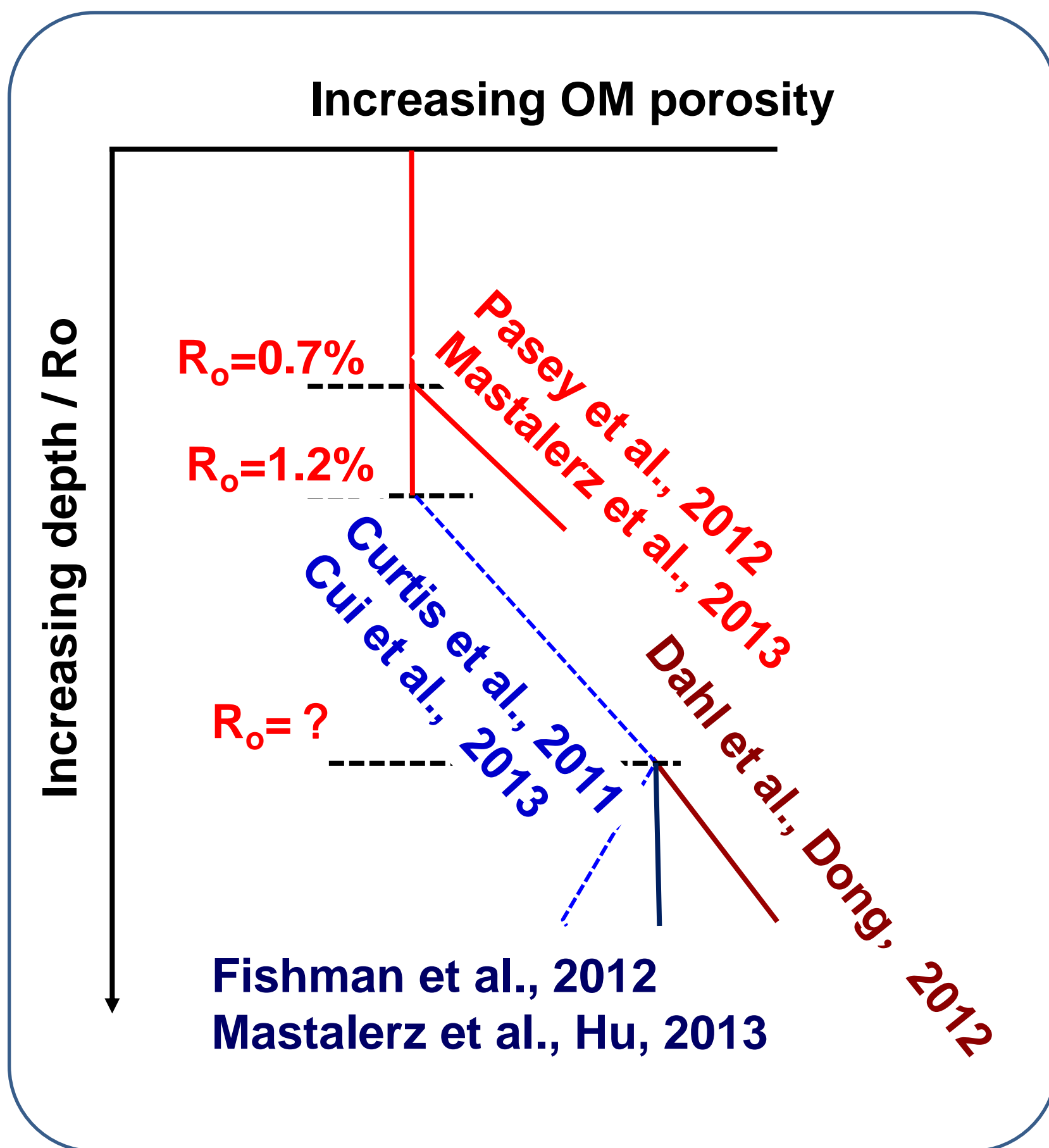
*Corresponding author email: wust@petrochina.com.cn



Abstract

With three different low mature shale samples as study object, the 3-D porosity evolution with temperature increase and its main controlling factors are analyzed based on the physical modeling under high temperature & pressure and nano-CT scanning data. More and more nano-pores were developed in organic-rich shale with the increase of maturity. The porosity calculated from the nano-CT scanning model increased from 0.56% to 2.06%, more than 250% times larger, when temperature increased from 20 ° C to 550 ° C. The process of porosity evolution can be divided into three phases. Firstly, porosity decreased rapidly from immature to low mature stage because of weak hydrocarbon generation and strong compaction; Secondly, porosity increased rapidly when the maturity increased from low mature stage to mature and post-mature stage, organic matter cracked into hydrocarbon (HC) massively, and clay minerals transformed intensively; Thirdly, porosity system kept stable when the shale entered into post-mature stage and the intensity of both HC generation and clay mineral transformation decreased. Organic matter thermal evolution, clay mineral transformation and brittle mineral transformation make different contribution to the porosity of shale, and the ratio is 6:3:1 respectively. It is inferred abundant organic matter pores occur when *Ro* is over 1.2%.

Scientific Problem



Problem: OM porosity evolution character & controlling factors

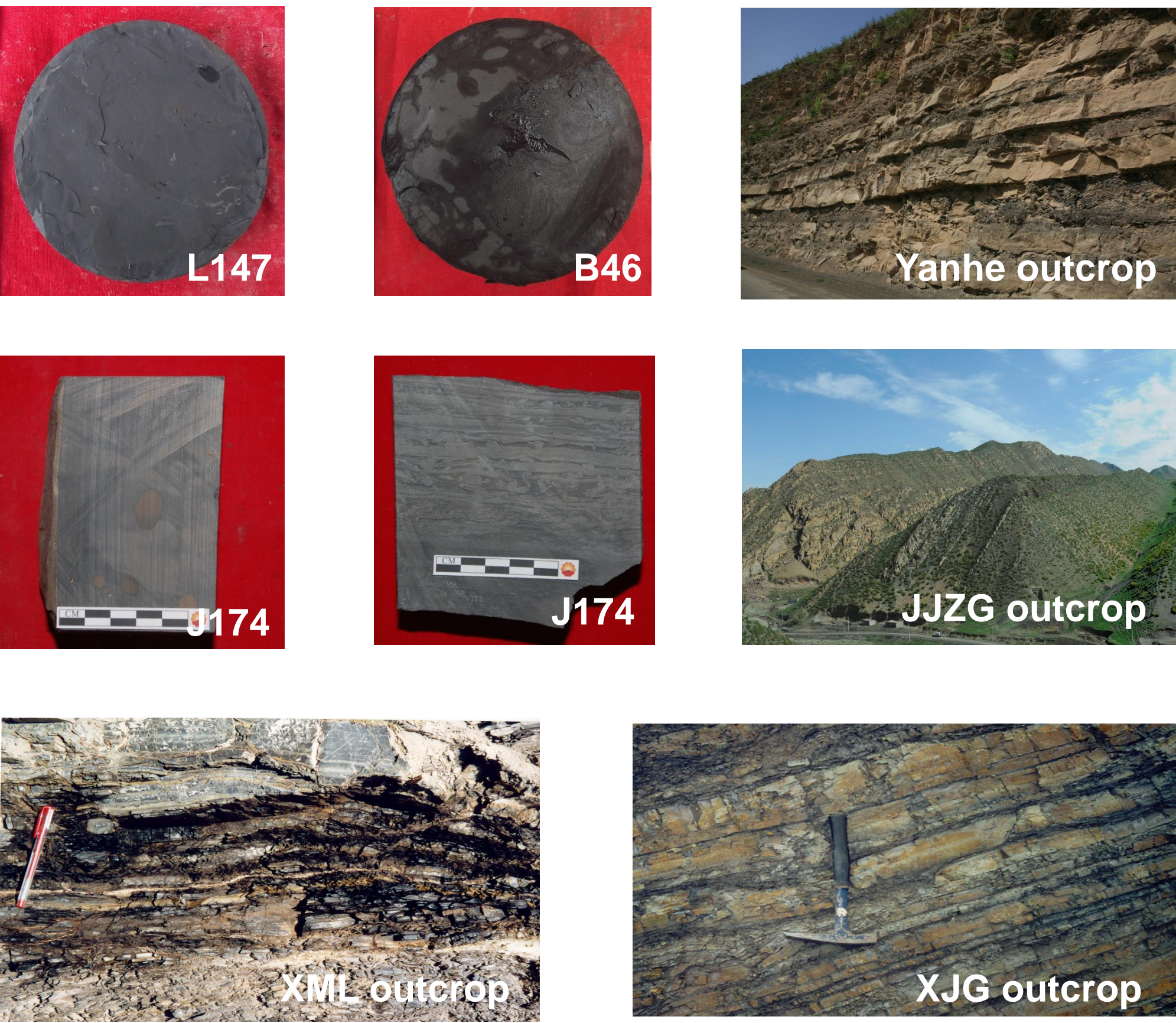
Different opinions on OM porosity evolution with increasing maturity

- Threshold of maturity for OM porosity is **0.7%? or 1.2%?**
- OM porosity increases continuously with maturity
 - Dahl et al., 2012; Dong, 2012
- OM porosity increases to a given maturity, then keeps stable
 - Fishman et al., 2012; Mastalerz et al., 2012
- OM porosity increases to a given maturity, then decreases with higher maturity
 - Curtis et al., 2012; Cui et al., 2012

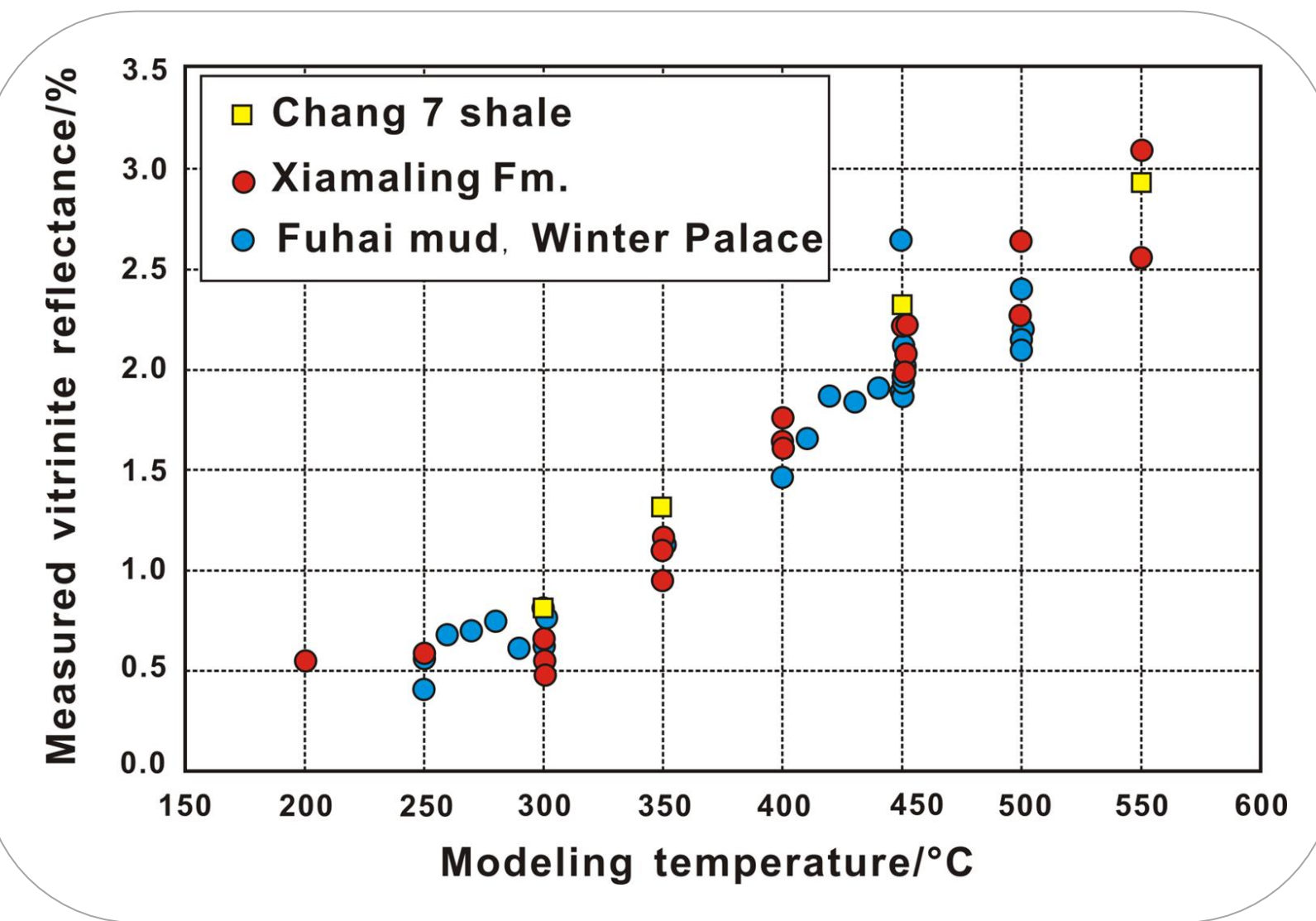
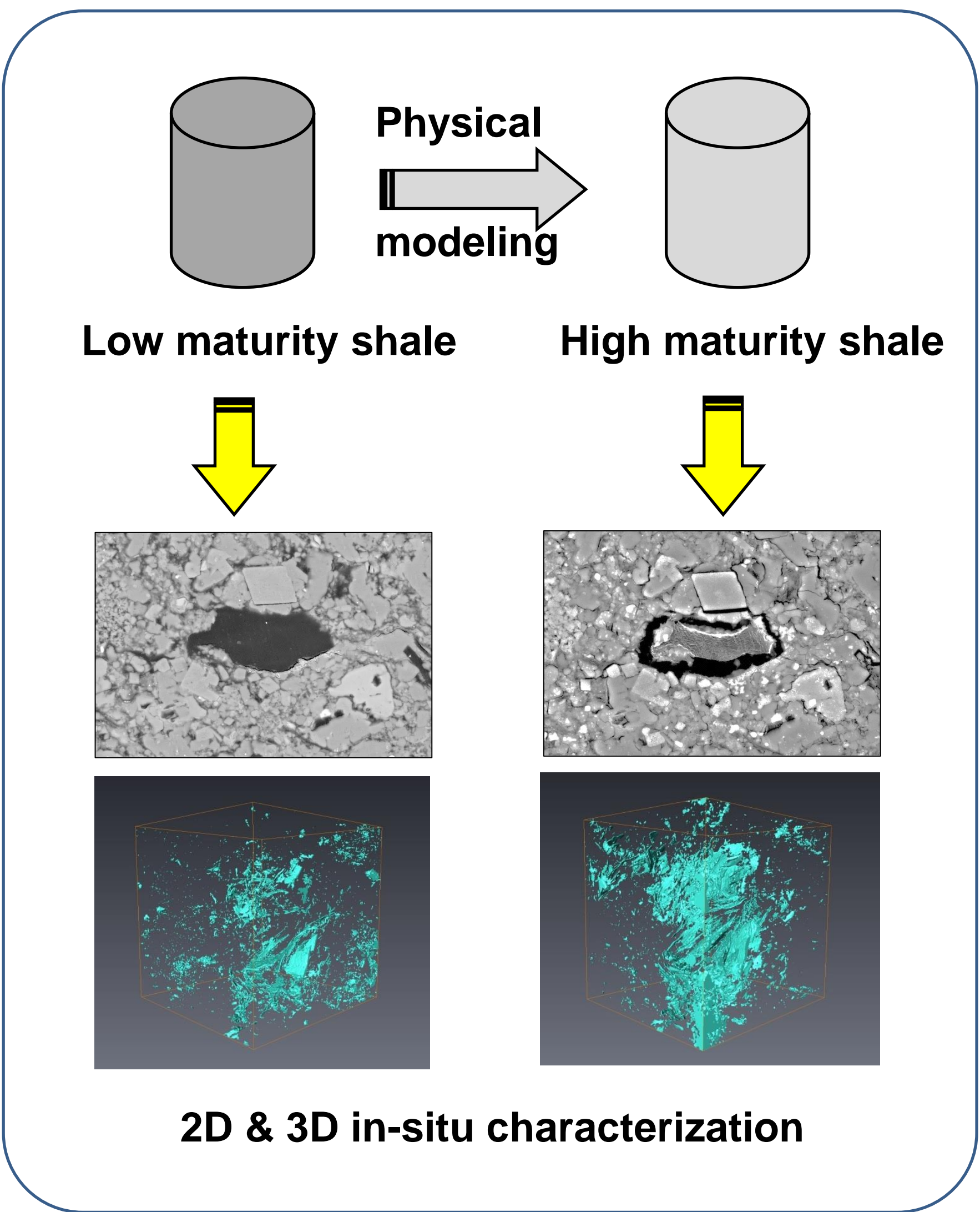
Sample Information

Information of three samples in the study

Type	Lacustrine shale with low salinity	Lacustrine shale with high salinity	Marine shale
Basin	Ordos Basin, Central China	Junggar Basin, NW China	North China
Formation	Upper Triassic Yanchang Fm.	Upper Permian Lucaogou Fm.	Middle Proterozoic Ximaling Fm.
Ro (%)	0.67	0.70	0.48
Kerogen	II ₁	II ₁	II ₁
TOC (%)	2.23	9.23	6.80
S ₁ (mg/g TOC)	5.11	1.4	1.14
S ₂ (mg/g TOC)	10.73	62.46	27.70
Tmax (°C)	426	444	434



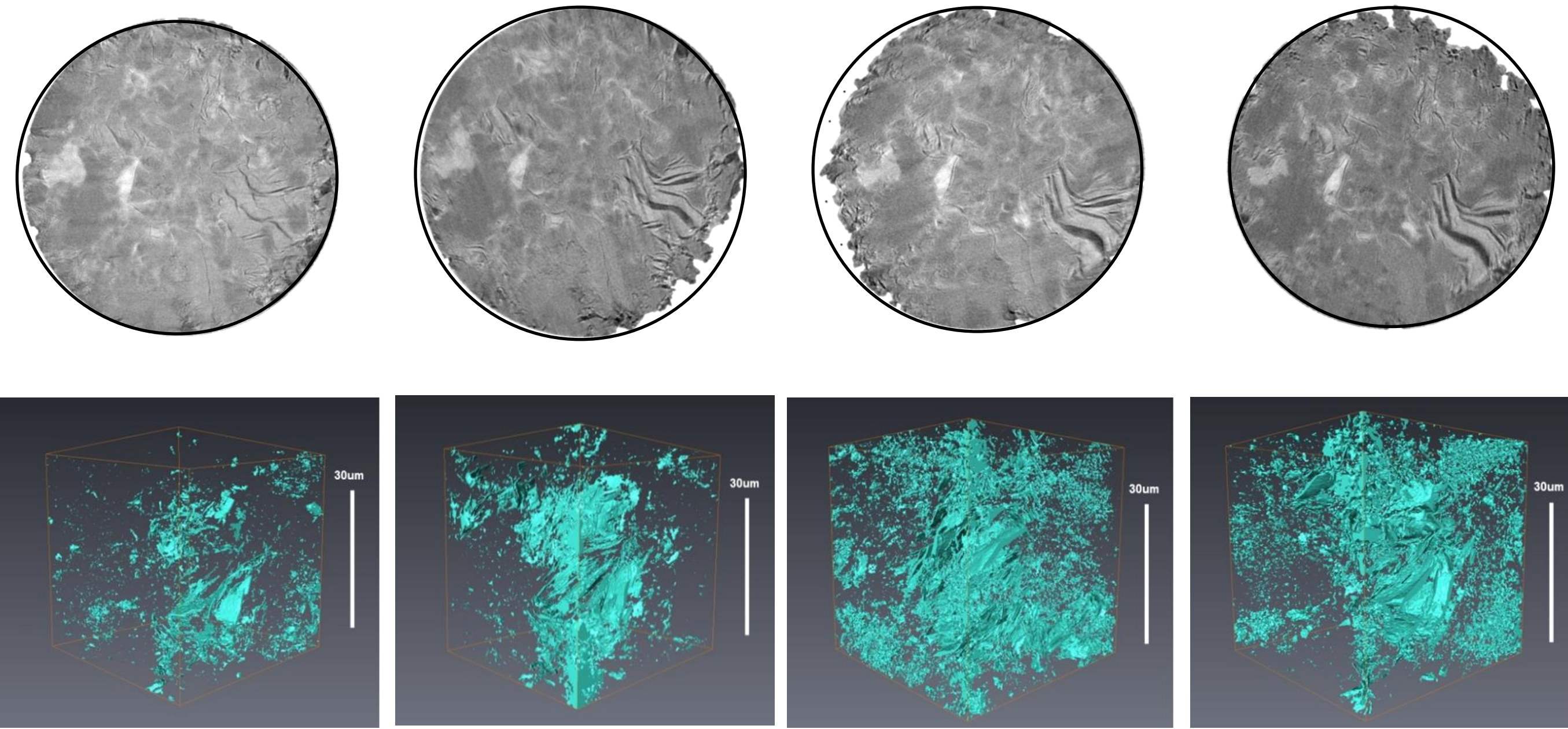
Method & Equipment



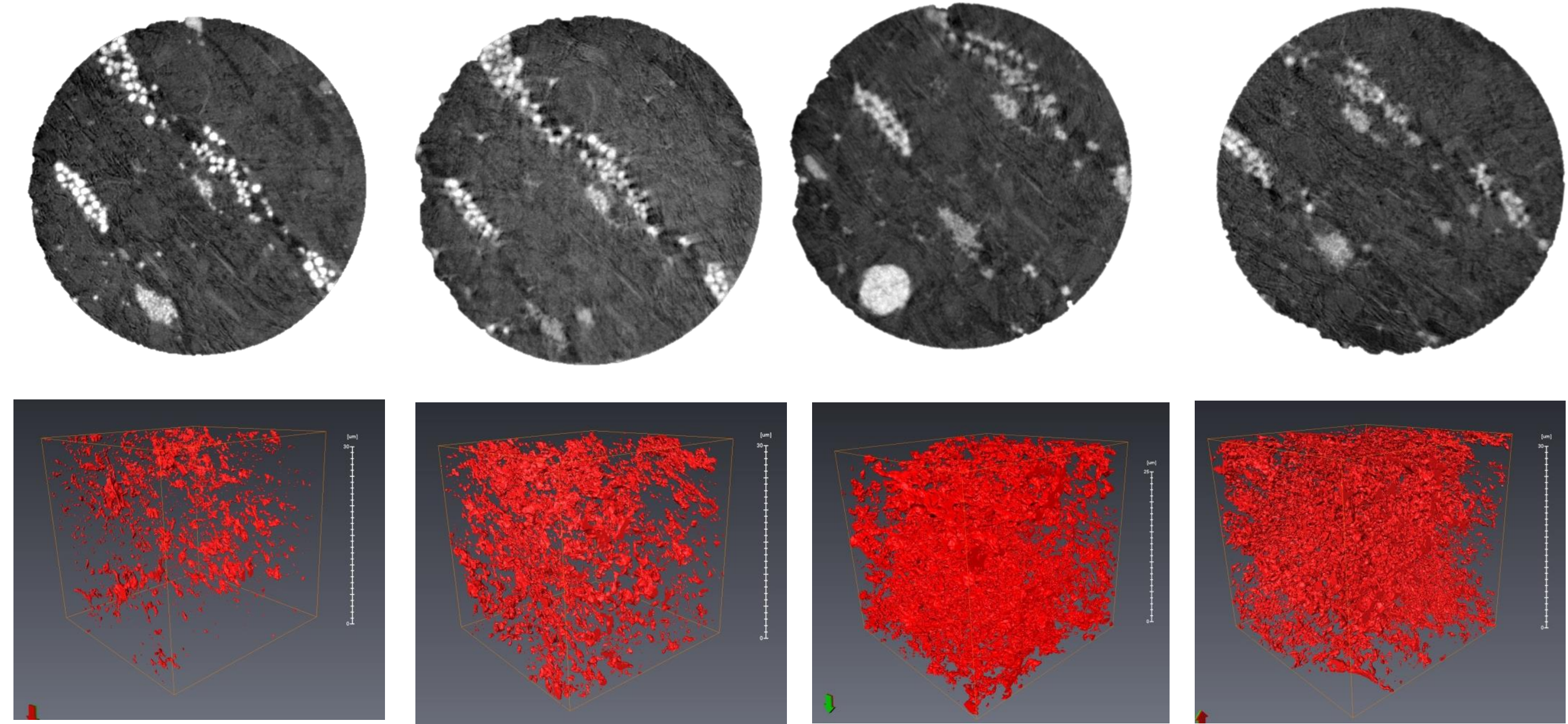
In this study, we establish the relationship between our modeling temperature and measured vitrinite reflectance, which provide reference for the parameter setting. The temperature points set in this study were 350 °C, 450 °C and 550 °C, corresponding to *Ro* of around 1.0%~1.5%, 2.0%~2.5% and 2.5%~3.0%, and mature-high mature, over mature and over high mature stages respectively.



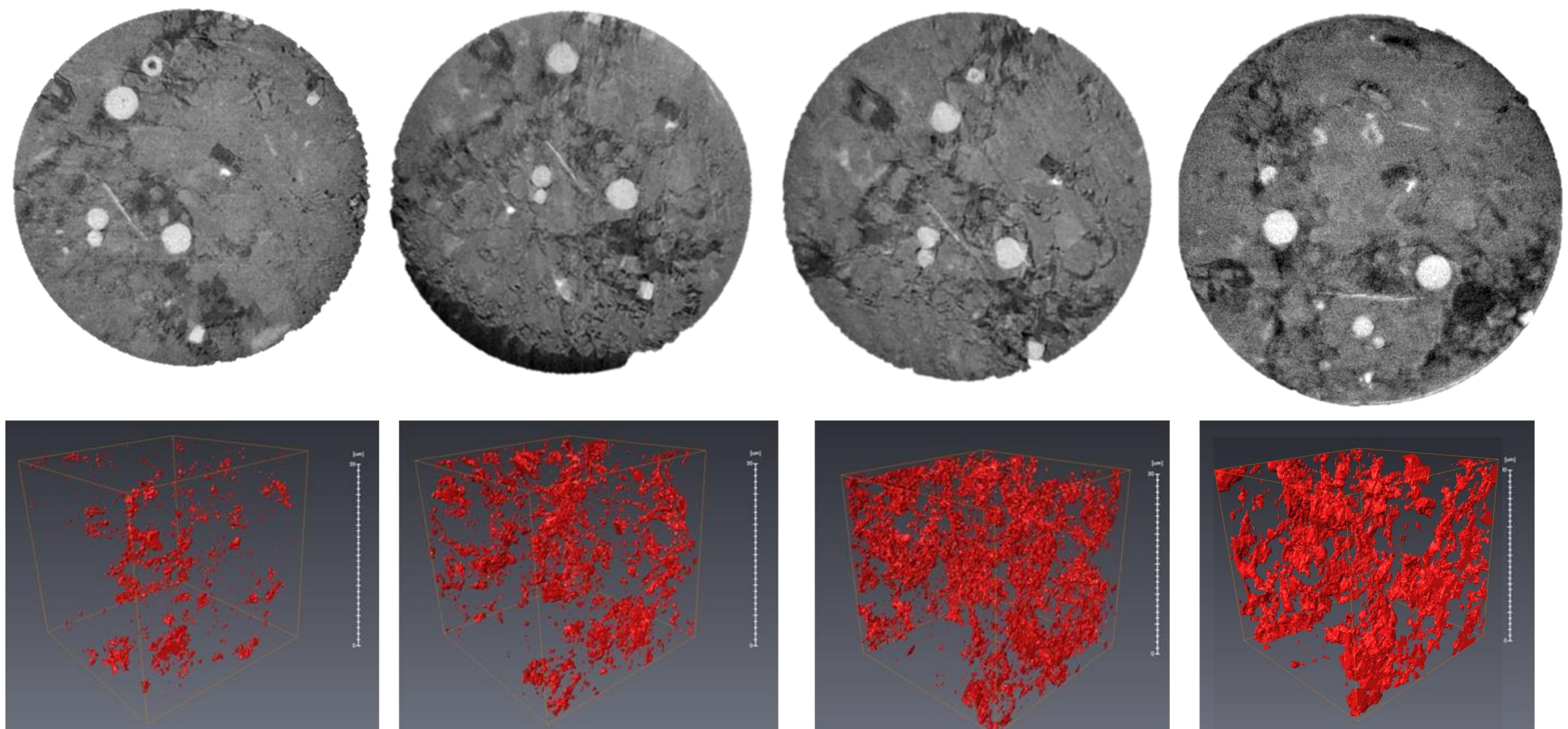
Nano-CT 3D Porosity Model



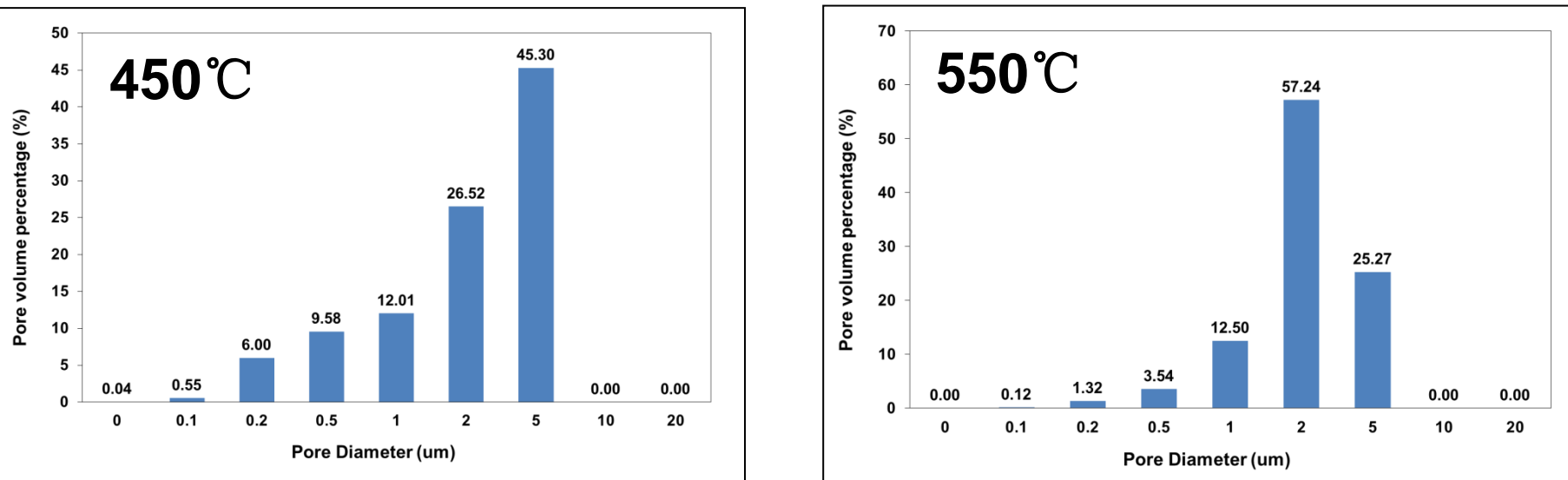
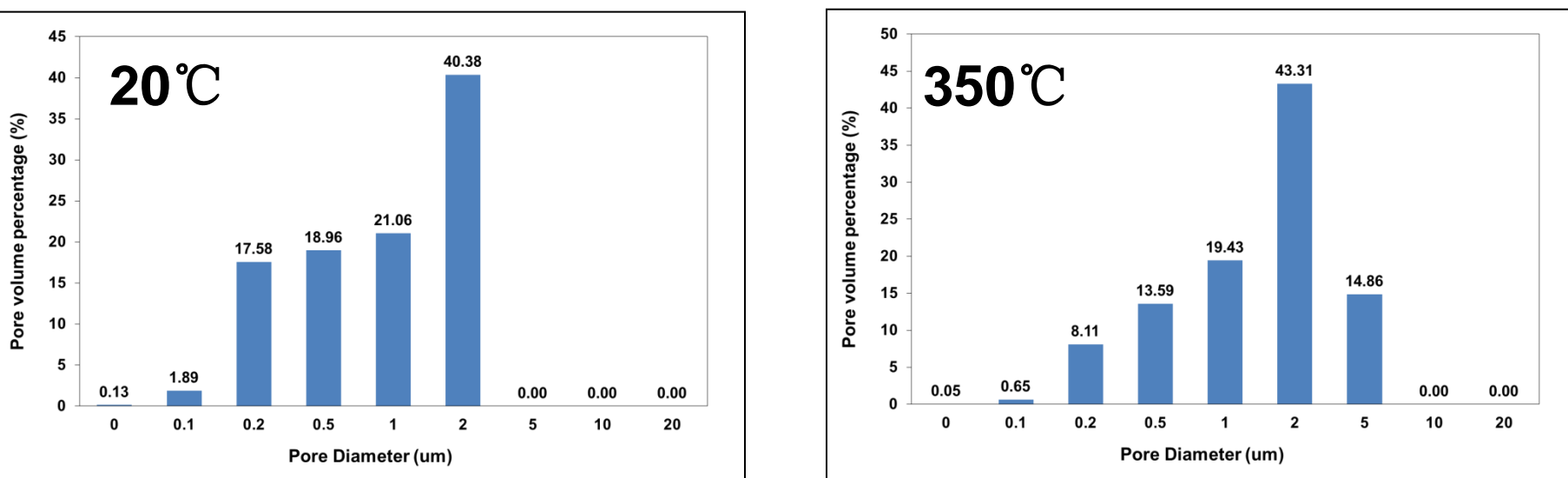
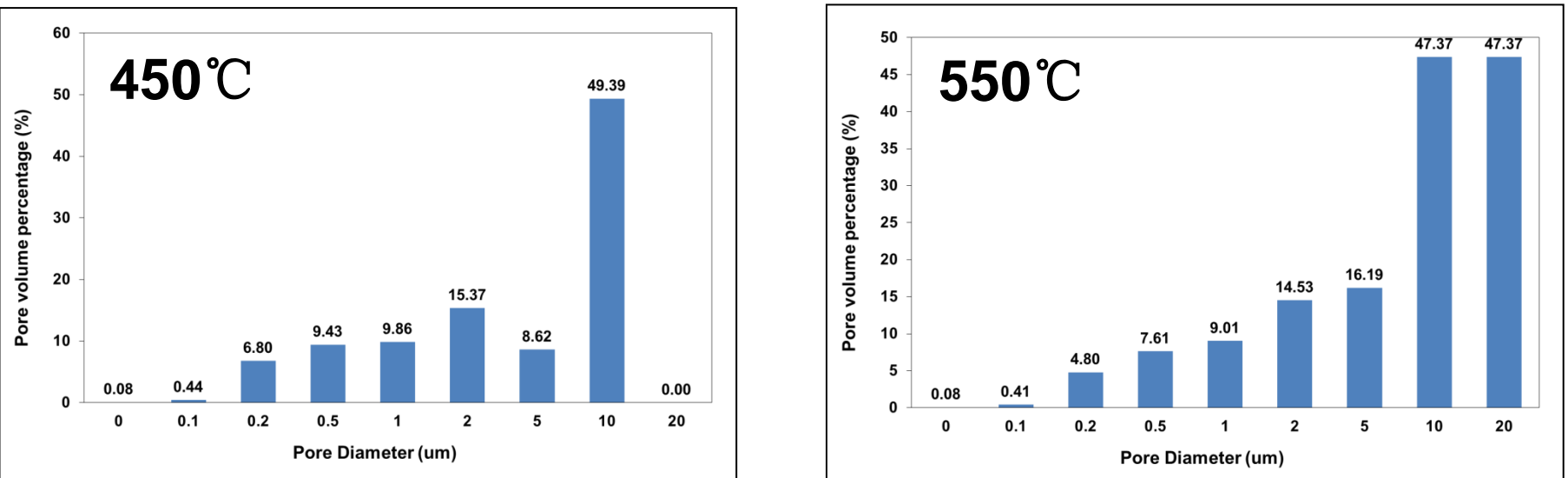
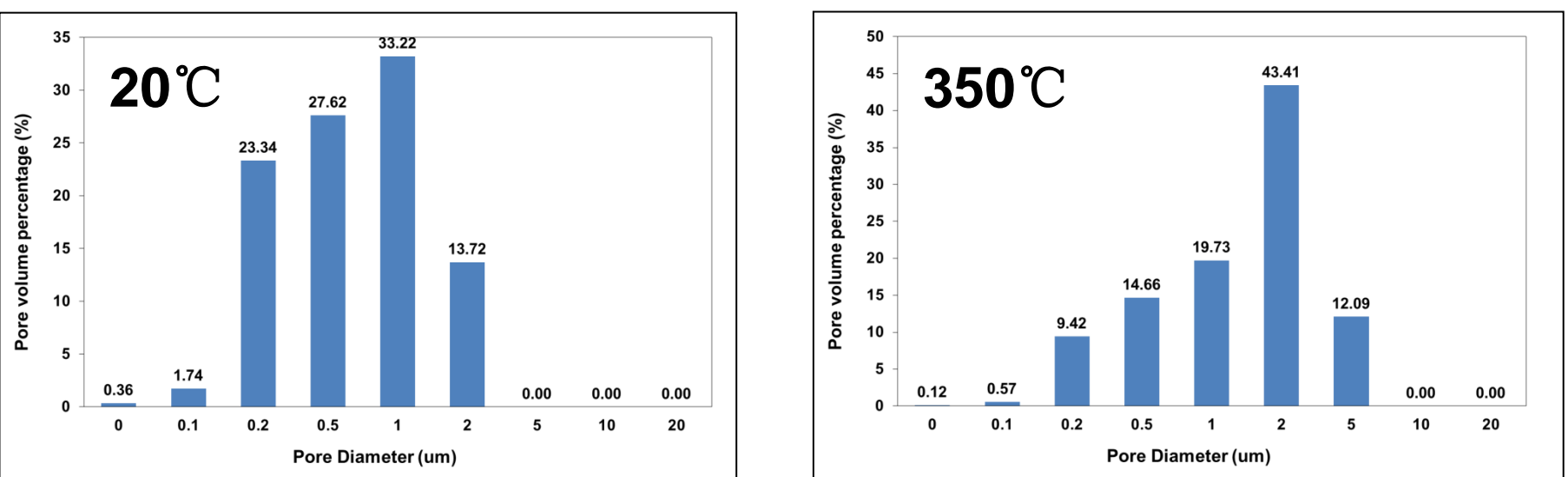
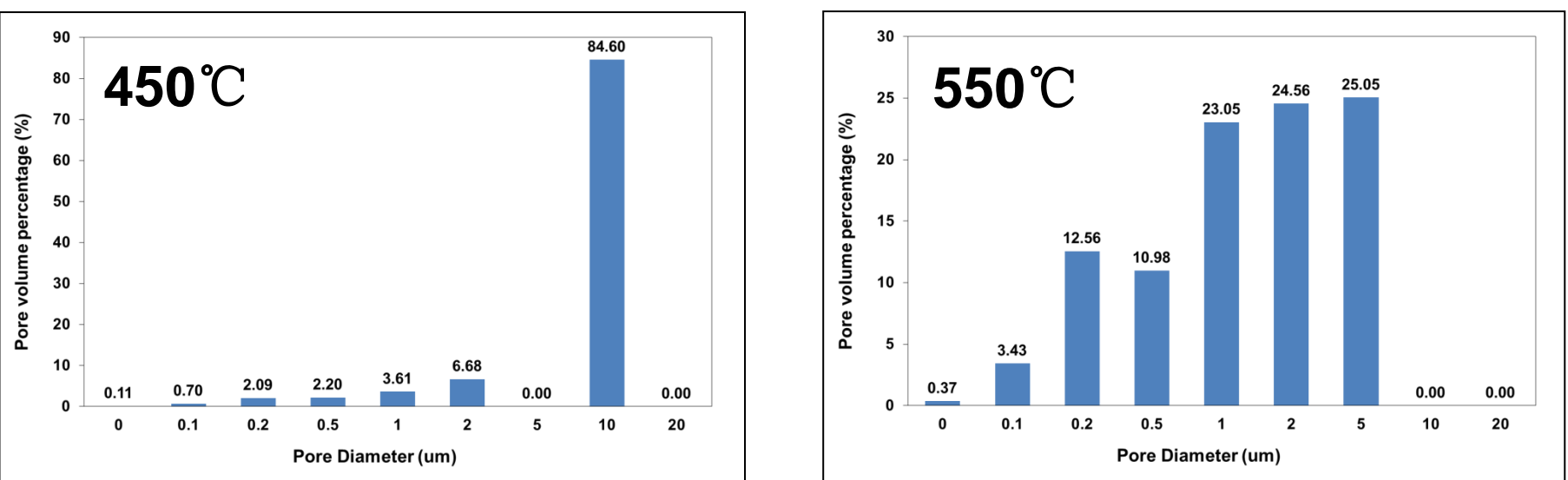
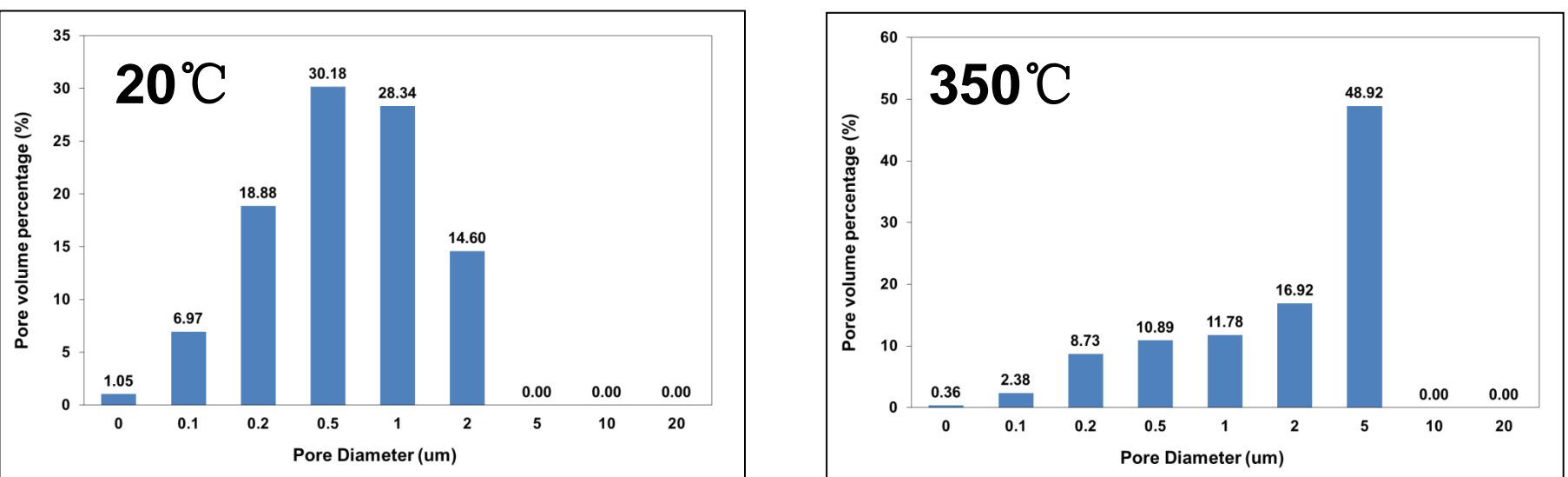
01: Chang 7 member, Yanchang Fm., Upper Triassic



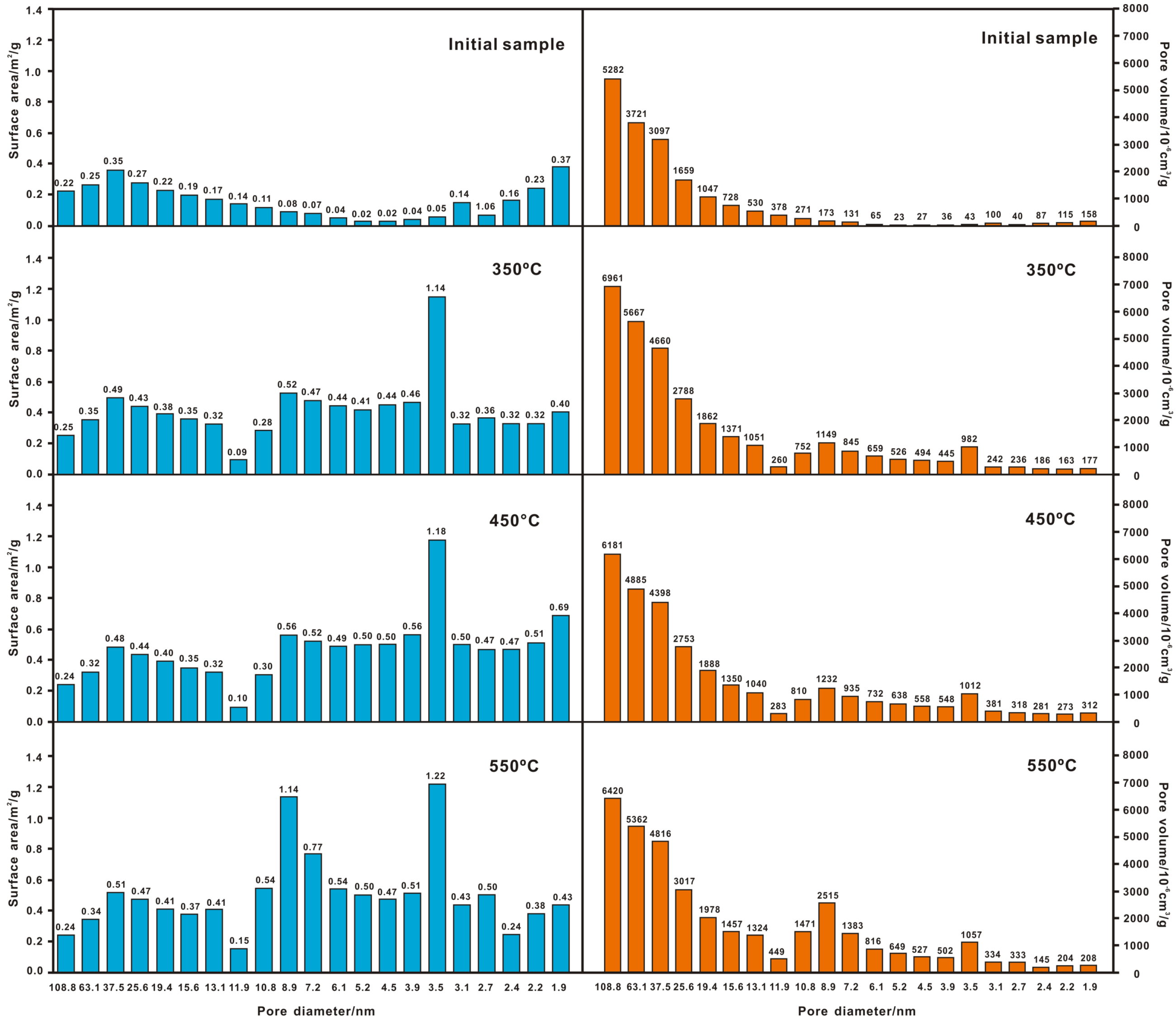
02: Lucaogou Fm., Upper Permian



03: Xiamaling Fm., Middle Proterozoic

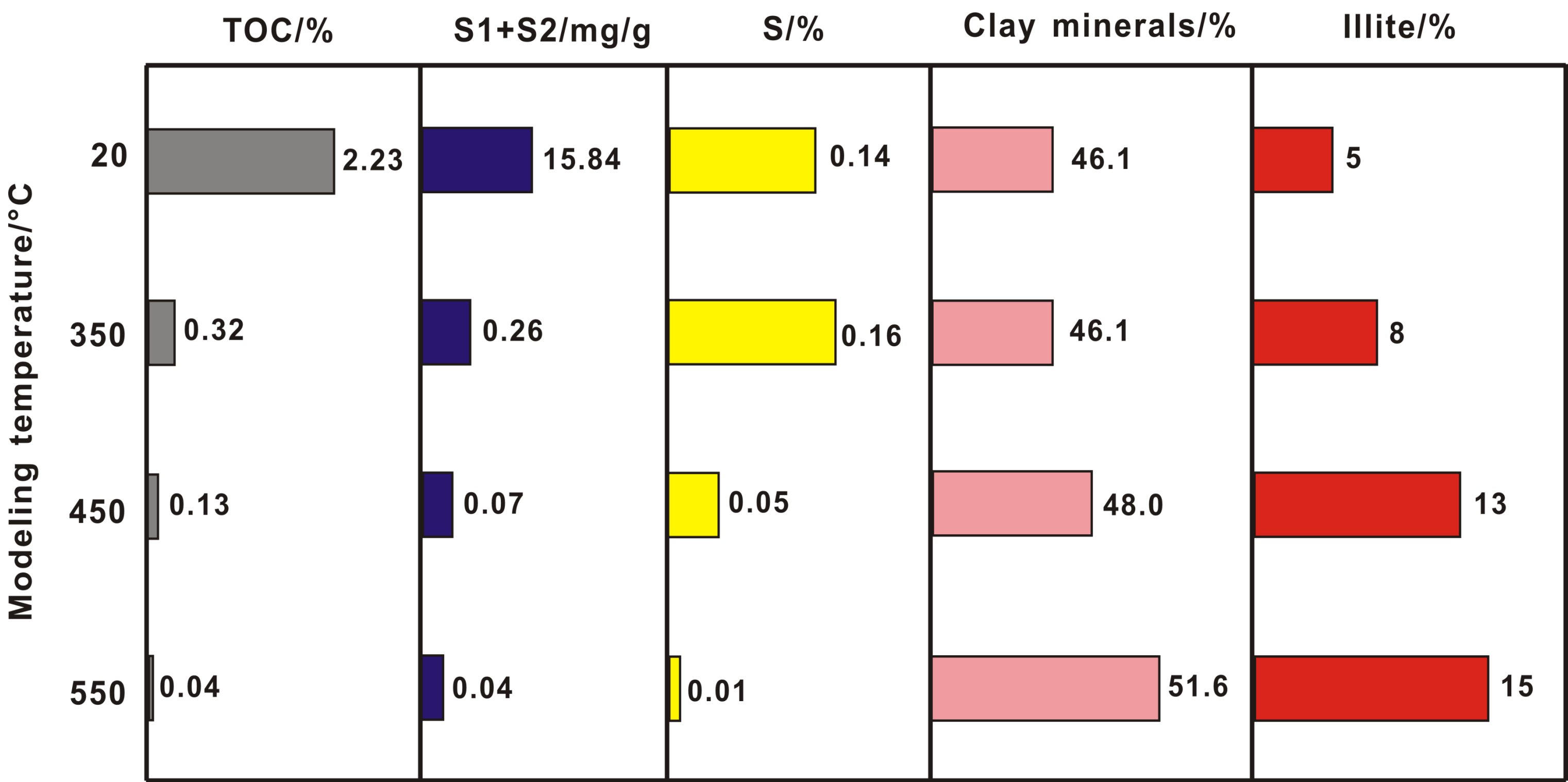


Nitrogen Adsorption Evaluation



- Nitrogen desorption result indicates with the rise of temperature, the pore system increases on the whole.
- Two peaks occur in specific surface area distribution histogram, the first peak occurring at 350 °C corresponds to the pore diameter of 3.5 nm, the second peak occurring at 550°C corresponds to the pore diameter of 8.9 nm. The two peaks are also reflected in the pore volume distribution histogram, indicating that the newly generated pores in the thermal evolution process are mainly 3 nm~10 nm in diameter.
- 350 °C (corresponding to Ro of 1.0%~1.5%, and oil and gas generation by thermal degradation-wet gas generation by thermal degradation stage) is the key temperature for the evolution of pores less than 100 nm in diameter.

Geochemical & Mineralogy Analysis

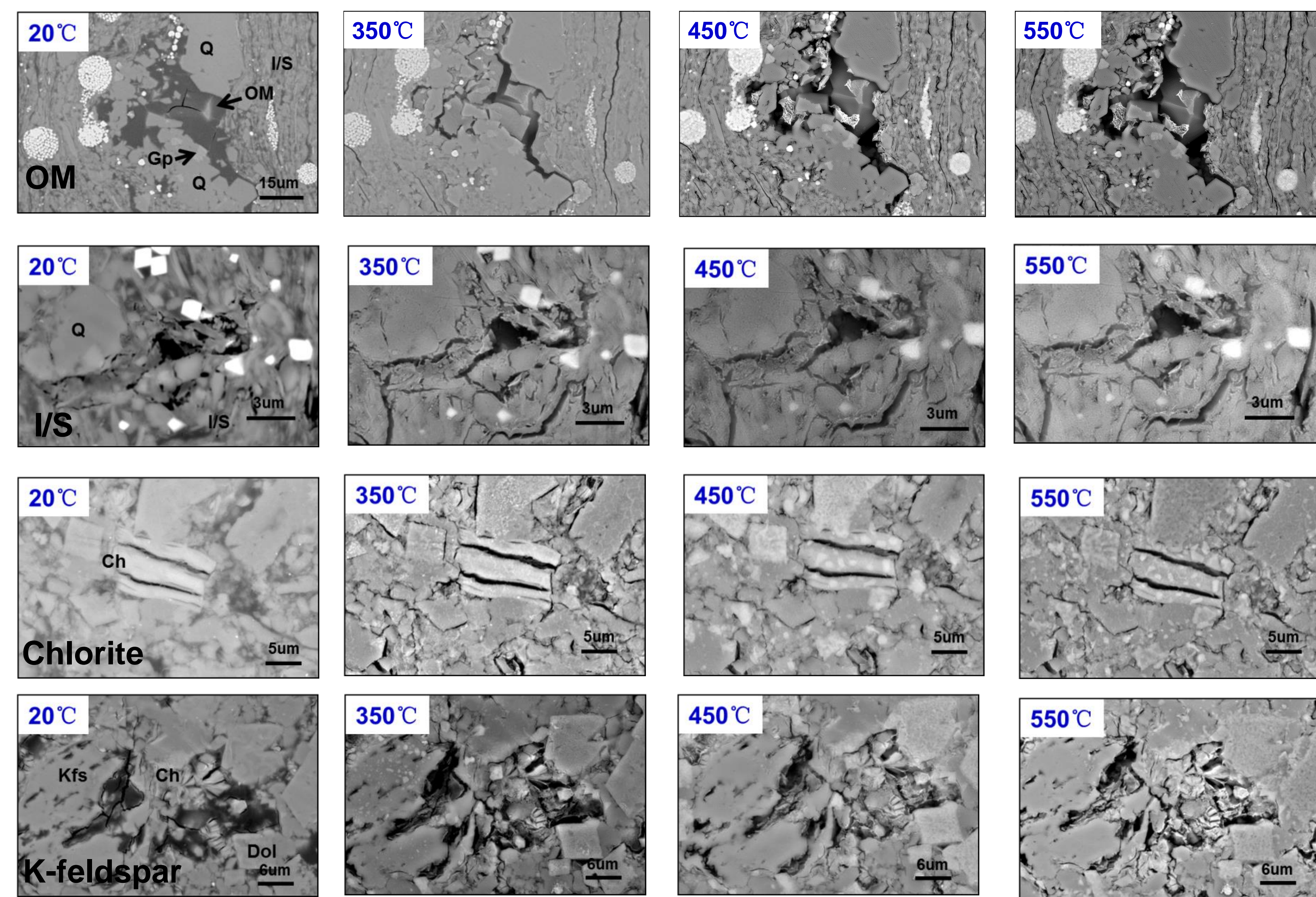


Chang 7 member, Yanchang Fm., Upper Triassic

- With the rise of modeling temperature, TOC decrease from 2.23% to 0.04%, (S1+S2) decrease from 15.84 mg/g to 0.04 mg/g, suggesting intense HC generation.
- XRD data shows that illite content increases from 5% to 15%, suggesting clay mineral transformation.
- All these data indicates that OM evolution, clay mineral transformation and porosity evolution take place at the same stage, which confirms the close relationship between them.

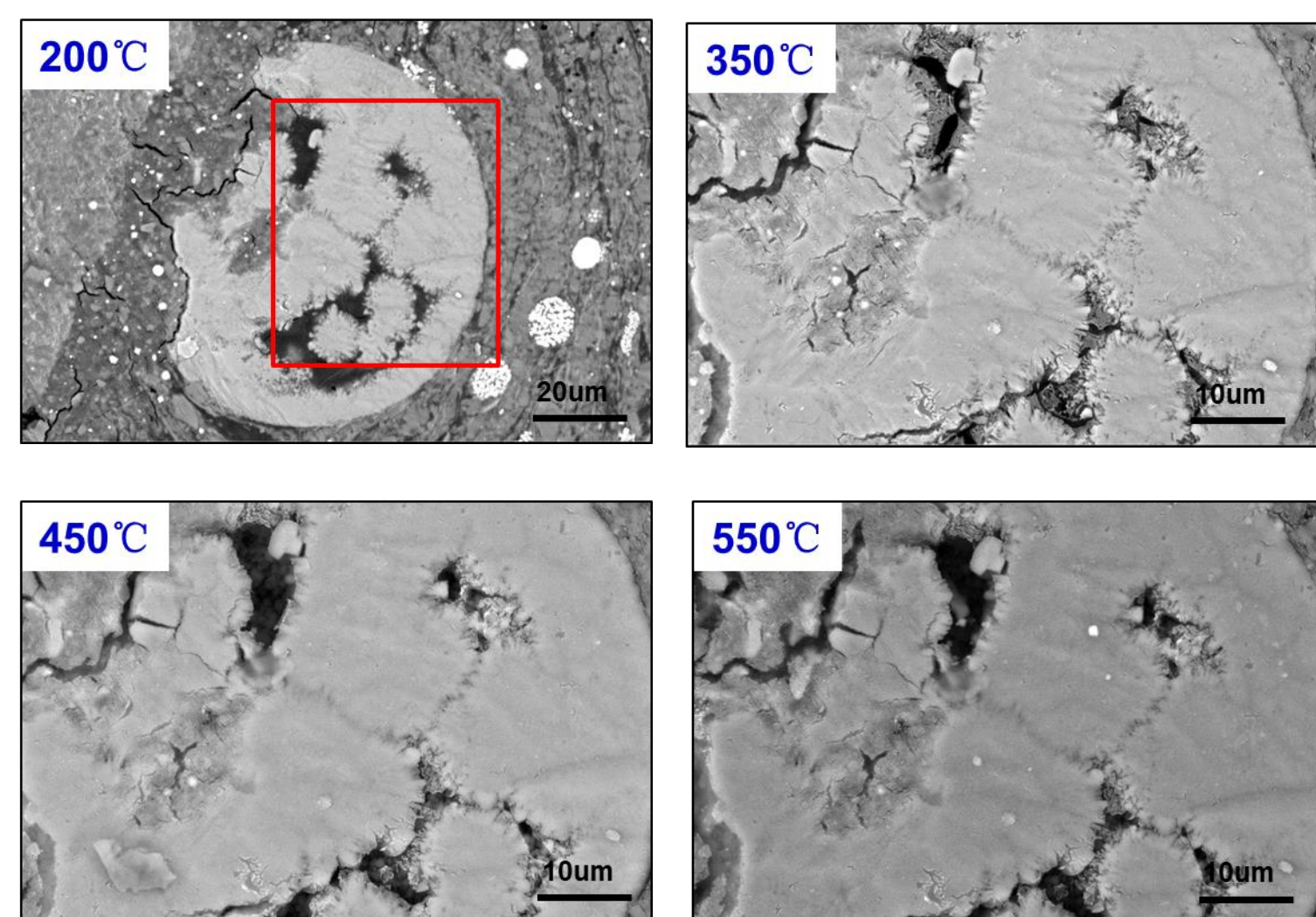
In-situ SEM Observation

Type 1: High Temperature & Low Pressure Experiment

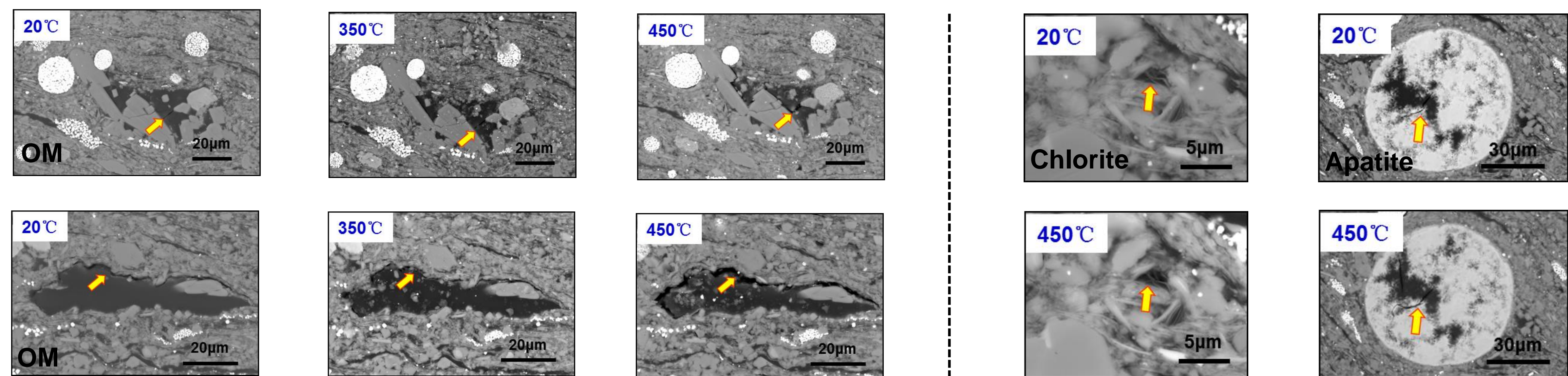


Organic matter thermal evolution, clay mineral transformation and brittle mineral transformation make different contribution to the porosity of shale, and the ratio is 6:3:1 respectively. It is inferred abundant organic matter pores occur when Ro is over 1.2%.

OM pore shapes at different temp.

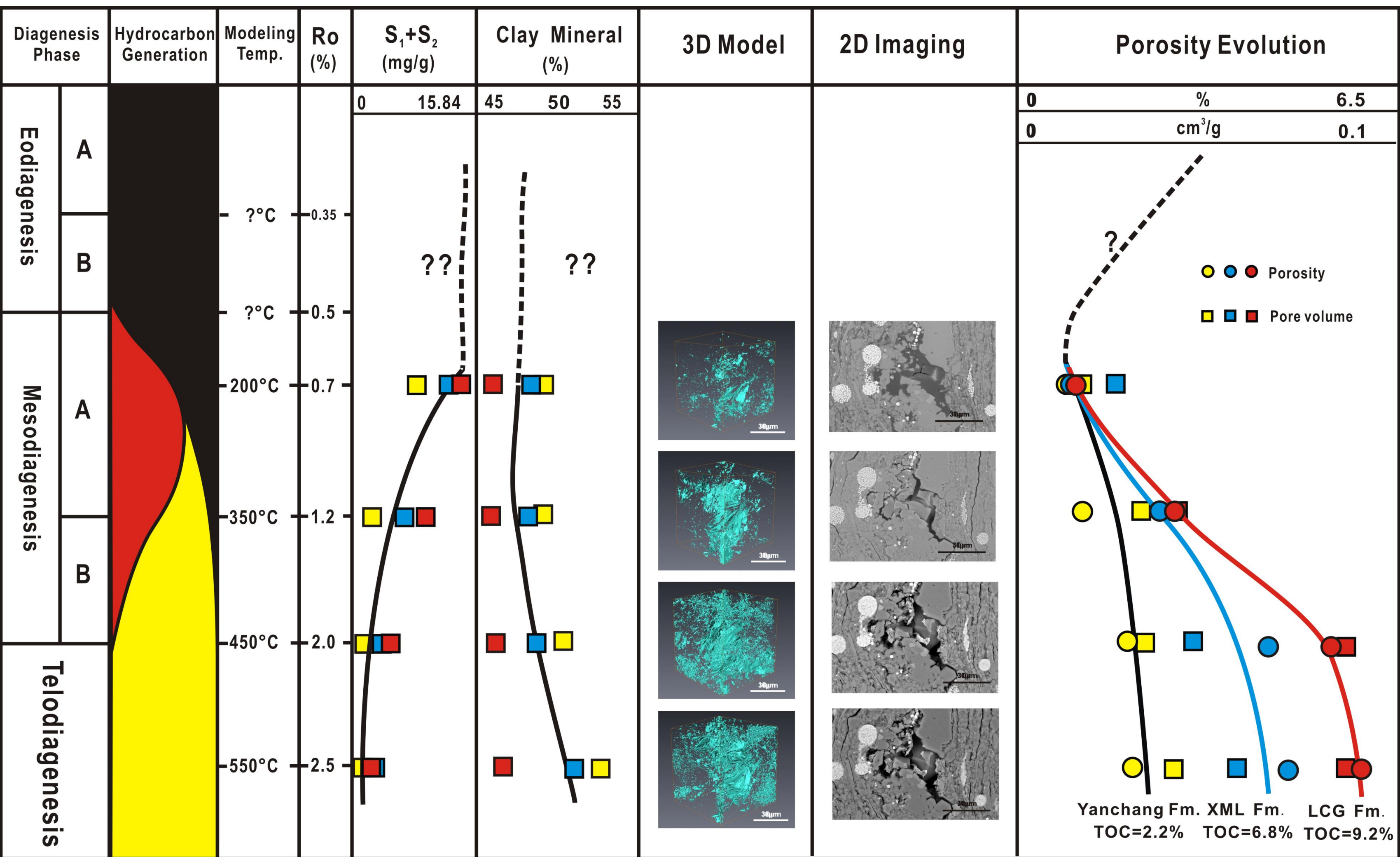


Type 2: High Temperature & High Pressure Experiment



At 90 MPa, HC generation, clay mineral transformation and brittle mineral evolution can generate new pores, among which HC generation raises porosity most obviously. Under certain temperature and pressure, organic matter cracks into HC, which is associated with the formation of elongated pores inside organic matter and along mineral matrix that will enlarge into new storage space.

Porosity Evolution Model



- Firstly, porosity decreased rapidly from immature to low mature stage because of weak HC generation and strong compaction
- Secondly, porosity increased rapidly when the maturity increased from low mature stage to mature and post-mature stage, organic matter cracked into hydrocarbon (HC) massively, and clay minerals transformed intensively
- Thirdly, porosity system kept stable when the shale entered into post-mature stage and the intensity of both HC generation and clay mineral transformation decreased.

Conclusions

- Shale porosity evolution is positively related with maturity. In this study, with the increase of organic matter maturity, nano-pores in organic-rich shale increase constantly.
- Different components make different contribution to porosity evolution of shale. OM thermal evolution: clay mineral transformation: brittle mineral transformation= 6:3:1.
- The porosity evolution process can be divided into three stages: first, porosity decreases rapidly from immature to low mature; then, porosity increases rapidly at oil & gas window; finally, porosity keeps stable at over-mature stage.
- Based on physical modeling at high T&P and nano-CT/SEM characterization, in-situ porosity evolution study can reconstruct 2-D and 3-D porosity models of the same sample at different maturity stages, the results of which can be compared by using 2-D fine point imaging and nitrogen adsorption, geochemical tests etc.

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