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Vitrinite Reflectance Suppression of the Triassic Chang 7 Shale: A Case Study from the Nijiahe Outcrop in the Ordos Basin

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ABSTRACT

The maturity of the Chang 7 shale in the Niejiahe section of the Tongchuan area of the Ordos Basin was studied based on Rock–Eval parameters, vitrinite reflectance, sterane isomerisation parameters, and aromatic methylphenanthrene parameters. The reflectance of vitrinite is 0.55% - 0.64%, and the value of Tmax is from 429°C to 453°C, which indicate the Chang 7 shale from the Niejiahe section is in the immature-low mature stage. While the isomerization parameters of C29 steranes reflect the maturity interval from 0.7% to 0.8%, and the equivalent vitrinite reflectance calculated from the methyl phenanthrene ratio of Chang 7 shale is from 0.74% to 0.86%, both of which indicate the Chang 7 shale has reached the mature stage. These reflect the significant suppression of vitrinite reflectance in the Chang 7 shale. There is a significant negative correlation between the vitrinite reflectance and the content of chloroform asphalt “A” and HI, indicating that the suppression of vitrinite reflectance is influenced by the infection of bitumen on vitrinite and hydrogen-enriched vitrinite. Therefore, when evaluating the hydrocarbon generation potential of Chang 7 shale based on vitrinite reflectance, the suppression of vitrinite reflectance should be considered.

KEYWORDS

Vitrinite reflectance suppression, Chang 7 shale, Biomarkers, Ordos Basin.

1. INTRODUCTION

Accurately determining maturity is meaningful for hydrocarbon exploration. Vitrinite reflectance (Ro) determined by optical petrology is a common parameter used as an indicator of the thermal maturity of organic matter in sediments. The reflectance of vitrinite (Ro) refers to the ratio of the reflected light intensity on the polished surface of homogeneous vitrinite or matrix vitrinite in coal under oil immersion conditions to the vertical incident light intensity, and is an important basis for determining coal rank in coal petrology. Due to the significant chemical and evolutionary similarities between kerogen and coal, the determination of vitrinite reflectance has been widely applied in the petroleum industry since the 1960s to determine the maturity of dispersed organic matter in shale or other sedimentary rocks. Vitrinite reflectance is currently the only comparable indicator of organic matter maturity internationally, which can objectively reflect the organic matter maturity of source rocks since the Late Paleozoic era. It is the most effective indicator to reflect the thermal evolution of organic matter. However, when correctly identifying vitrinite in dispersed organic matter, there are two main shortcomings: the difficulty of measuring vitrinite reflectance and the suppression of vitrinite reflectance (Tang et al. 2016). The suppression of vitrinite reflectance is a well-known phenomenon, and if not identified, it may have a significant impact on the results of thermal history and resource evaluation, and seriously affect exploration decisions.

Accurately determining the reflectance is of great significance for shale oil evaluation. Normally, as maturity increases, the density and viscosity of shale oil gradually decrease(Liu et al. 2012), resulting in a higher movable oil content (Wang et al. 2019; Zhang et al. 2014). As the maturity increases, the proportion of light components (alkanes) in shale oil increases, and the differences between the retained hydrocarbon components in shale and the structure of krogen become more and more significant, resulting in a gradual decrease in their adsorption solubility(Wang et al. 2019). The discovery of terrestrial shale oil in Jiyang Depression shows that when Ro is greater than 0.9%, it is more favorable for shale oil exploration(Song et al. 2019). The Qingcheng oil field has been discovered in the Chang 7 shale in the Ordos Basin(Fu et al. 2020). Therefore, accurately determining the maturity of Chang 7 shale is of great significance for the resource evaluation and development of shale oil.

2. SAMPLES AND EXPERIMENTS

A total of 28 samples of the Chang 7 Member of Yangchang Formation were collected from Niejiahe profile in Ordos Basin, China. The location of Niejiahe profile is shown in Fig.1.

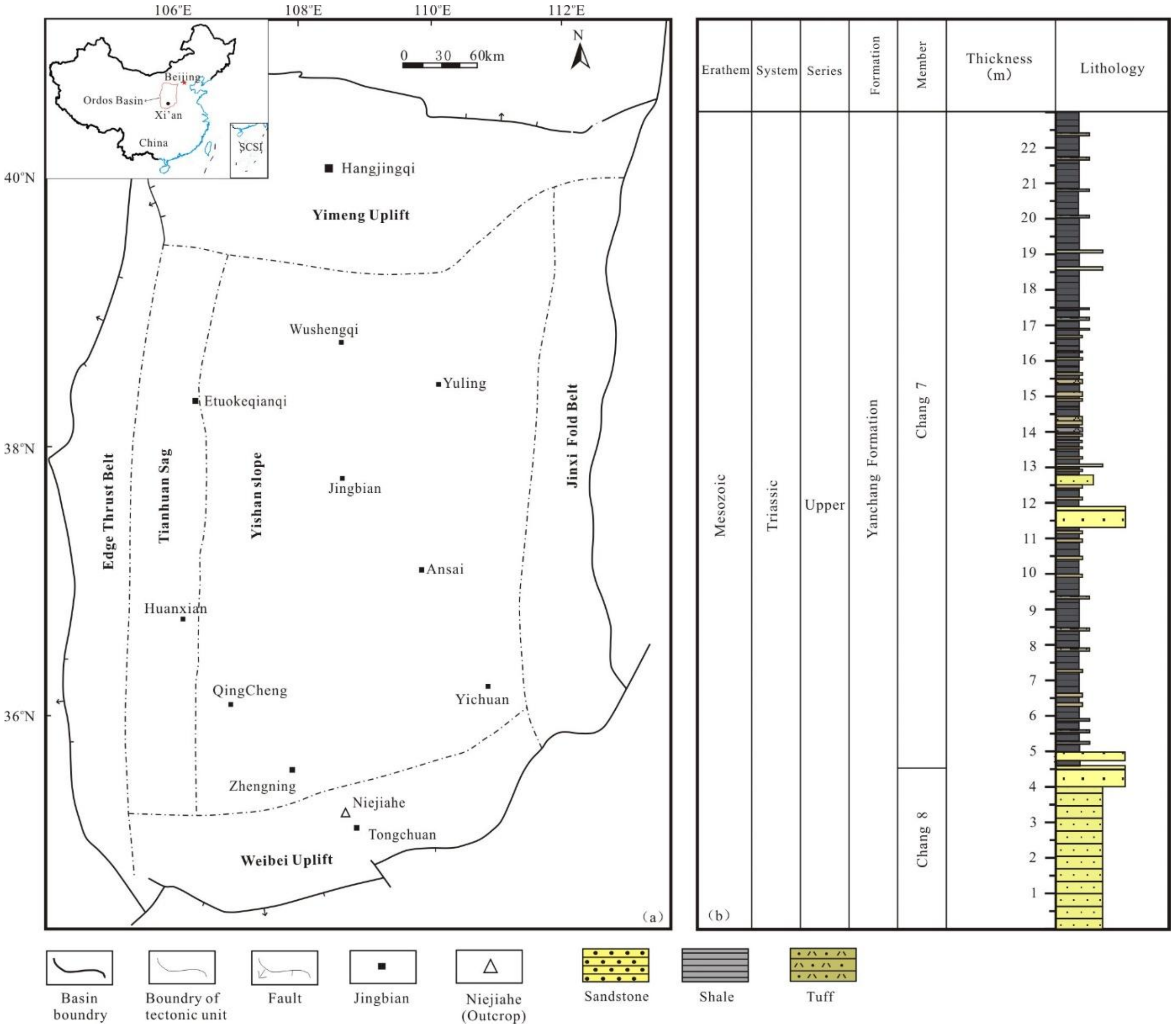

Fig 1. The location of Niejiahe outcrop (a) and the stratigraphic chart of the Niejiahe section (b).

Organic geochemical screening analyses were carried out to characterize the samples. The samples were analyzed for total organic carbon (TOC, wt.%) contents by combustion in an ELTRA CS-800 induction furnace, with HCl treatment of the samples to remove carbonate-bonded carbon before TOC determination. The samples were further pyrolyzed in a Source Rock Analyzer (SRA) system.

All the shale samples were also analyzed for HI and Tmax using routine Rock-Eval experiments performed on a Rock-Eval 6 instrument. Five shale samples were analyzed for vitrinite reflectance. Reflectance measurements (random, oil immersion) were performed on a QDI302 microspectrophotometer equipped with a Leica DM4500P polarizing microscope.

All rock samples were cleaned prior to powdering. Soxhlet extraction was conducted using chloroform/methanol (87:13) for 72 h and the isolated extractable organic matter was separated into saturated hydrocarbons, aromatic hydrocarbons and polars. Gas chromatographic mass spectrometry (GC–MS) analyses of the saturate fractions were performed with a HP6890GC/5973MSD instrument equipped with a HP-5MS fused silica column (30m × 0.25 mm i.d., film thickness 0.25μm). The GC oven temperature for analysis of the saturate fractions was initially held at 50°C for 2 min, programmed to 100°C at 20°C /min and to 310°C at 3°C/min, and held at 310°C for 16.5 min. Biomarker ratios were calculated from peak areas of individual compounds.

3. RESULTS AND DISCUSSION

3.1. Thermal maturity from Tmax

The Tmax is a widely used maturity indicator, while the relationship between petroleum generation zones and Tmax varies (Yang and Horsfield 2020). it is usually considered that the samples with Tmax value < 437°C are classified as being immature, whereas the Tmax value ranging from 437°C to 450°C is considered to be in low mature, the Tmax value ranging from 450°C to 480°C is considered to be in mature (oil window), respectively(Zhang and Li 2018). The Tmax values of lower member and upper member are in the range of 429°C to 453°C (average = 435°C). Which means that the Chang 7 shales are mainly in immature to low mature stage.

3.2. The maturity indicated by vitrinite reflectance

Previous studies have shown that there are abundant vitrinite in the Chang 7 shale, with a content of 5.13% to 21.95% (Pang et al. 2023). The content of vitrinite in the Chang 7 Member shales on the Niejiahe section range from trace to 10% (Table 1). Despite the low vitrinite content of most of the samples (Fig.2), according to the Chinese Standard SY/T 5124-2012, more than 20 data points was measured. On the Niejiahe section, the shale of Chang 7 Member is 18.5m thick. Without the influence of magma, they should have consistent thermal evolution maturity. As shown in Table 1, the vitrinite reflectance values of the other samples is between 0.55% and 0.64%, with an average of 0.58%. which means that the samples were in early the early oil generation stage.

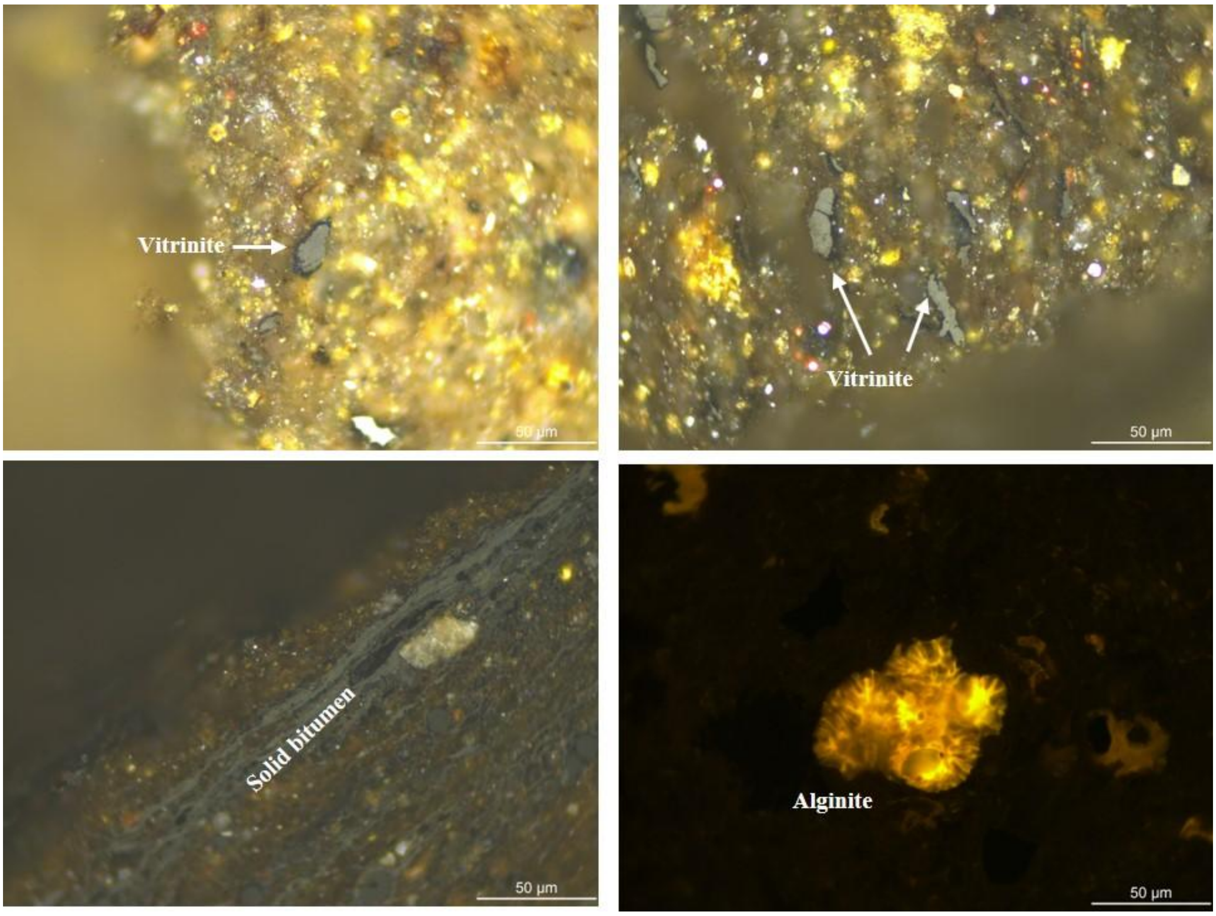

Fig. 2 Photomicrographs of typical vitrinite Chang 7 Shale in Niejiahe section

Table 1. The maceral composition, vitrinite reflectance and the content of Chloroform bitumen "A" of Chang 7 shale

Samples ID	Vitrinite (%)	Liptinite (%)	Inertinite (%)	Sapropelite (%)	Solid bitumen (%)	Ro (%)	Chloroform bitumen "A" (%)
NJH-21-02	trace	1	trace	5	94	0.58	0.8571
NJH-21-08	trace	1	/	3	96	0.55	1.1442
NJH-21-17	trace	1	trace	2	97	0.56	1.0811
NJH-21-27	trace	2	trace	2	96	0.55	1.2677
NJH-21-B1	10	/	/	/	90	0.64	0.1426

3.3. The maturity indicated by biomarkers and aromatic hydrocarbon

The C29 sterane isomerization ratios are common indicators for assessment of the thermal maturity of crude oils and sediments (Peters et al. 2005). The C29 ααα 20S / (20S + 20R) sterane and C29 αββ / (αββ + ααα) sterane ratios of the Chang 7 Member shale of Triassic Yangchang Formation appear have not reached their equilibrium ratios, with ratios in a range of 0.46–0.47 and 0.38–0.42, respectively, and average 0.46 and 0.39 (Table 2). This shows that the samples were in the early oil generation stage, with the equivalent vitrinite reflectance in a range of 0.7-0.8% (Fig. 3).

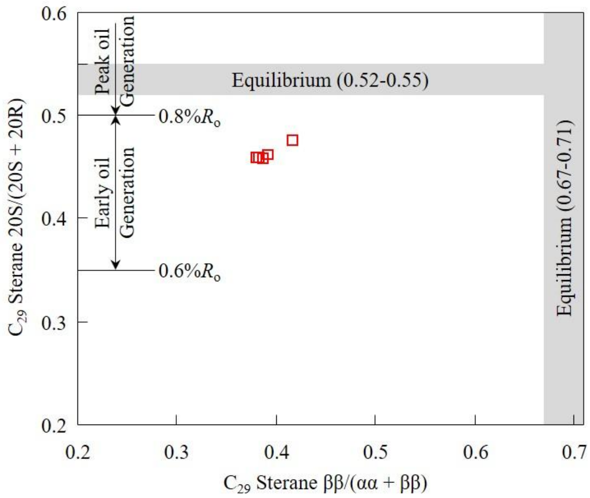

Fig. 3 C29 20S / (20S + 20R) versus C29 ββ / (ββ + αα) sterane ratios for source rock from the Niejiahe section(Hao et al. 2010).

The study samples contain abundant alkylphenanthrenes (Fig.4). Fig.4 shows a representative annotated chromatogram of phenanthrene and methylphenanthrene of the aromatic fraction form the Chang 7 shale samples. The methylphenanthrene index (MPI1) (MPI1=1.5(2-MP+3-MP)/(P+1-MP+9-MP)) is frequently applied to calculate the equivalent vitrinite reflectance (%Rc) of source rock and oil (%Rc1 = 0.44 + 0.55 × MPI1, Rc<1.3%; %Rc2 = 0.60MPI1+0.40, Rm<1.35%(Chen et al. 2022)). The MPI1 values of the study samples have a range of 0.53-0.60 (averaging 0.57). The calculated %Rc1 and %Rc2 values range between 0.73–0.77 and 0.75–0.79, respectively, and average 0.75 and 0.77 (Table 2).

Another common aromatic parameter based on the distribution of methylphenanthrenes is methylphenanthrene distribution fraction (F1=(2-MP+3MP)/(2-MP+3-MP+1-MP+9-MP) and F2=2-MP/(2-MP+3-MP+1-MP+9-MP)) proposed by Kvalheim et al. (1987). They pointed out ratios of F1 and F2 show a linear positive relationship with Ro within oil window maturity, and have two empirical formulas: Ro=-0.166+2.242F1 and Ro=-0.112+3.739F2 (Kvalheim et al. 1987). The empirical formulas have been used for the maturity valuation of crude oil and source rock extensively (Chen et al. 2023; Quan et al. 2017; Song et al. 2019). The calculated vitrinite reflectance (calculated from F1 and F2) of Chang 7 shale ranges from 0.78 to 0.86 and from 0.74 to 0.82, respectively (Table 2)

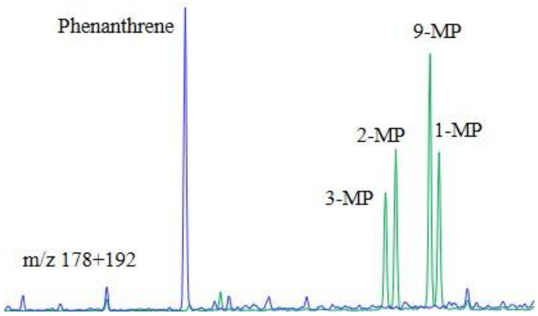

Fig. 4 Partial mass chromatograms (m/z 178 and m/z 192) of the aromatic hydrocarbon fractions showing the distributions of phenanthrene and methylphenanthrene in source rock from the Niejiahe section.

Table 2 The C29 sterane isomerization ratios and the aromatic maturity parameters of Chang 7 shale from the Niejiahe section

Samples ID	C29-20S/20S+20R	C29-ββ/αα+ββ	MPI 1	%Rc1	%Rc2	F1	F2	%Rc -F1	%Rc -F2
NJH-21-02	0.46	0.39	0.53	0.73	0.75	0.41	0.25	0.81	0.82
NJH-21-08	0.46	0.38	0.59	0.76	0.78	0.41	0.23	0.80	0.75
NJH-21-17	0.46	0.39	0.60	0.77	0.79	0.40	0.23	0.79	0.74
NJH-21-27	0.46	0.39	0.56	0.75	0.76	0.40	0.23	0.78	0.74
NJH-21-B1	0.47	0.42	0.58	0.76	0.78	0.43	0.24	0.86	0.79

3.4. The suppression of vitrinite reflectance

From the comparison of vitrinite reflectance, C29 sterane isomerization ratios, and aromatics maturity parameters, the maturity reflected by the vitrinite reflectance is significantly lower the biomarkers and methylphenanthrene, and there may be some degree of suppression of vitrinite reflectance. Previous studies have shown that the adsorption/infection of hydrocarbons or asphaltenes generated by hydrogen rich components (amorphous, algal, and crustal) by vitrinite can lead to a decrease in vitrinite reflectance(Hutton and Cook 1980; Peters et al. 2018). It can be clearly seen from Figure 5 that there is a significant negative correlation between the reflectance of vitrinite and the content of chloroform asphalt "A", indicating that hydrocarbons have a significant suppression effect on the reflectance of vitrinite in the Chang 7 shale of the Niejiahe section.

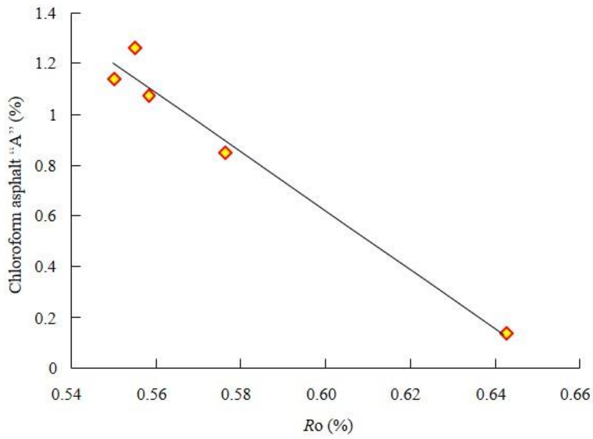

Fig. 5 The cross plot of Ro and Chloroform asphalt "A" of Chang 7 shale from the Niejiahe section

In addition, under strong reduction conditions, anaerobic bacteria are particularly developed, causing biochemical degradation of organic matter, consuming oxygen in the organic matter and forming relatively hydrogen-enriched humic components (precursors of hydrogen-enriched vitrinite), thereby reducing its reflectivity(Newman and Newman 1982). The Chang 7 shale was deposited in a strongly reducing environment(Fu et al. 2018) and has the conditions to form hydrogen-enriched vitrinite. Hydrogen index (HI) can indicate the relative degree of hydrogen enrichment in the maceral components, and it can be seen from Figure 6 that the reflectance of vitrinite gradually decreases with the increase of hydrogen index. This indicates that hydrogen-enriched vitrinite in the Chang7 shale of the Niejiahe section has a significant suppression effect on vitrinite reflectance.

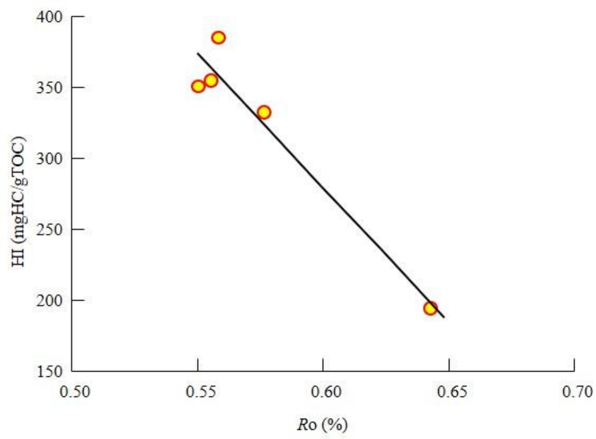

Fig. 6 The cross plot of Ro and HI of Chang 7 shale from the Niejiahe section

Previous studies have shown that shale with high natural gamma, hydrogen index, and TOC often experiences suppression of Tmax(Snowdon 1995). The Chang 7 shale exhibits significant natural gamma rays(Liu et al. 2021), high hydrogen index, and TOC. From Figure 7, the Tmax value of Chang 7 shale from the Niejiahe section gradually decreases with the increase of HI. The samples from the Niejiahe section with 18.5m thick should have an approximate Tmax value, while the values are in the range of 429°C to 453°C, which also indicates the suppression effect of hydrogen-enriched maceral on the maturity.

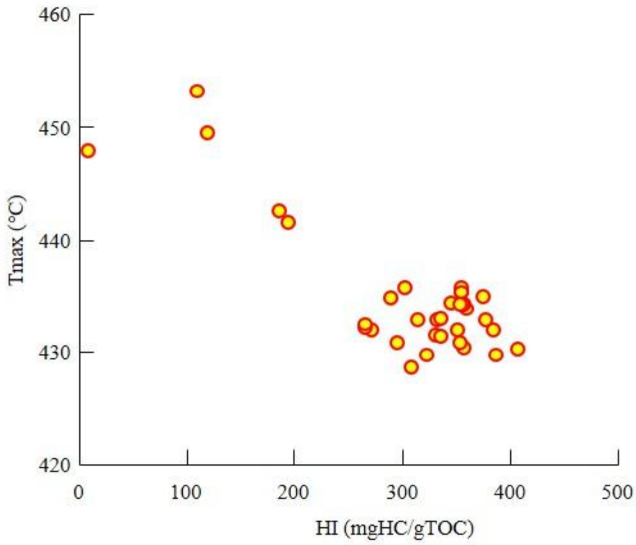

Fig. 7 The cross plot of Tmax and HI of Chang 7 shale from the Niejiahe section

3.5. Significance

Maturity is of great significance for shale oil resource evaluation. The vitrinite reflectance of the Chang 7 shale in the Qingcheng oil field of the Ordos Basin is 0.7% -1.1%(Fu et al. 2020), which has achieved success in shale oil exploration, while the equivalent vitrinite reflectance of its crude oil based on the methylphenanthrene isomerization ratio is 0.8% -1.3%(Wang 2018), indicating that there may also be a certain degree of vitrinite reflectance suppression in the Chang 7 shale in the Qingcheng area. Due to the abundant shale oil and the strongly reducing environment of the Chang 7 shale(Fu et al. 2018), the suppression phenomenon may be also caused by the adsorption/infection effect of hydrocarbons and the hydrogen-enriched vitrinite.

Through the above analysis, it can be found that there may be a common phenomenon of suppression of vitrinite reflectance in the Chang 7 shale of the Ordos Basin. Therefore, whether for conventional crude oil or shale oil, resource evaluation based on vitrinite reflectance may underestimate the hydrocarbon generation potential of Chang 7 shale. Therefore, when evaluating the hydrocarbon generation potential of Chang 7 shale, attention should be paid to the influence of vitrinite reflectance suppression.

4. CONCLUSION

(1) Based on vitrinite reflectance, C29 sterane isomerization ratios, and aromatic methylphenanthrene, it is found that the Chang 7 shale in the Ordos Basin exhibits significant suppression of vitrinite reflectance.

(2) There is a significant negative correlation between the reflectance of vitrinite and chloroform asphalt "A" and HI, indicating that bitumen infection and hydrogen-enriched vitrinite jointly lead to the occurrence of suppression phenomenon of vitrinite reflectance.

(3) When evaluating the hydrocarbon generation potential of Chang 7 shale in the Ordos Basin, attention should be paid to the influence of vitrinite reflectance suppression.

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