

Article

Correction of Light and Heavy Hydrocarbons and Their Application in a Shale Oil Reservoir in Gaoyou Sag, Subei Basin—A Case Study from Well SX84

Qi Zhi ¹, Shuangfang Lu ^{2,3,*}, Pengfei Zhang ⁴, Hongsheng Huang ¹, Junjie Wang ¹ and Zizhi Lin ¹¹ School of Geoscience, China University of Petroleum (East China), Qingdao 266580, China² Sanya Offshore Oil & Gas Research Institute, Northeast Petroleum University, Sanya 572025, China³ Key Laboratory of Continental Shale Hydrocarbon Accumulation and Efficient Development, Ministry of Education, Northeast Petroleum University, Daqing 163318, China⁴ College of Earth Science and Engineering, Shandong University of Science and Technology, Qingdao 266590, China

* Correspondence: lushuangfang@nepu.edu.cn

Abstract: To accurately evaluate the shale oil resources in the Funing Formation of the Gaoyou Sag, Subei Basin, light and heavy hydrocarbon correction models of S_1 were developed based on the rock pyrolysis of liquefrozen, conventional, and oil-washed shales. The improved $\Delta\log R$ technique was applied to establish the TOC, S_1 , and S_2 logging evaluation methods. The results showed that the S_2 values after oil washing were significantly lower than before. The difference between these two S_2 (ΔS_2) values is the heavy hydrocarbon correction amount of S_1 , which is about 0.69 S_2 . There was almost no loss of light hydrocarbons during liquefrozen shales' pyrolysis tests; the ratio of liquefrozen to conventional S_1 values is the light hydrocarbon correction factor, which is about 1.67. The corrected S_1 is about 3.2 times greater than the conventional shale-tested value. The S_1 and TOC are obviously "trichotomous"; a TOC greater than 1.5% and corrected S_1 larger than 4.0 mg/g corresponds to the enriched resource. The logging estimated results show that the total shale oil resources in the E_1f_2 of the Gaoyou Sag are about 572 million tons, of which the enriched resource is about 170 million tons.

Keywords: light and heavy hydrocarbon correction; classified resource; resource evaluation; shale oil; Gaoyou Sag



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1. Introduction

Unconventional oil and gas have been of worldwide concern for oil and gas exploration. Based on the "thermal triangle" theory, conventional and unconventional resources in the 25 basins in North America were evaluated, showing that the recoverable resources of unconventional resources far exceeded the conventional resources [1]. Although this method is imprecise, it can be seen that unconventional oil and gas resources have excellent prospects. Meanwhile, shale oil has received more attention for unconventional energy exploration in the world.

Shale oil refers to the oil contained in a shale series dominated by shale, including the oil in shale pores and fractures, as well as the oil in the adjacent layers and interlayers of tight carbonate or clastic rocks [2,3]. Shale oil is the hydrocarbon resource formed in a mature shale series (including mudstone and shale), mainly occurring in the free and adsorbed states, but with few dissolved states. It is characterized by continuous distribution, considerable resources, and a long production cycle [4,5].

In recent years, shale oil exploration in North America has developed rapidly and commercial production of shale oil has been achieved in the United States. There have been significant shale oil discoveries in central Alberta and southern Texas, as well as other favorable areas such as the Los Angeles region, the south of the Gulf of Mexico, and eastern

Canada Shale oil development has significantly increased oil production in the United States and Canada [6]. According to the U.S. EIA, shale oil production will increase to 7.1×10^6 barrels per day by 2040 [7]. Moreover, shale oil shows huge exploitation potential in China, such as in the Ordos, Sichuan, Songliao, Subei, and Bohai Bay basins [8–11].

Resource assessment is essential in shale oil exploitation. The organic matter in shale consists of two parts: extractable organic matter (shale oil) and non-extractable organic matter (kerogen). Shale oil includes hydrocarbon and non-hydrocarbon NSO compounds. Chloroform-extractable bitumen “A” is most similar to shale oil in composition, with hydrocarbons and NSO compounds. However, most of the C_{6-13} light hydrocarbons are vaporized during the volatilization of chloroform in the chloroform-extractable progress.

S_1 represents the fraction of the hydrocarbons in shale oil and is a standard parameter used to evaluate the shale oil content. S_1 refers to the volatilized hydrocarbons during the rock pyrolysis analysis when temperatures are less than $300\text{ }^{\circ}\text{C}$, which are C_{7-33} hydrocarbons. However, shale light hydrocarbons (C_{6-13}) have primarily dissipated due to the long placement time [12]. S_2 represents the generated hydrocarbons from kerogen when the temperature gradually rises from $300\text{ }^{\circ}\text{C}$ to $600\text{ }^{\circ}\text{C}$ during the rock pyrolysis. However, some heavy hydrocarbons already present in the shale have boiling points much higher than $300\text{ }^{\circ}\text{C}$. Moreover, the adsorption and swelling of kerogen cause it to be impossible for the residual hydrocarbons to be completely volatilized before $300\text{ }^{\circ}\text{C}$ [13], corresponding to this residual hydrocarbon being detected as S_2 . Therefore, due to the storage conditions of the cores and experimental testing techniques, S_1 lost some light and heavy hydrocarbons, resulting in S_1 being significantly lower than the actual in situ shale oil content. Thus, the light and heavy hydrocarbon correction for S_1 is required to evaluate the total shale oil content.

Shales exhibit low porosity and permeability, causing shale oil development to be difficult, and not all resources can be extracted efficiently [14–17]. Under the current economic and technological conditions, only part of the shale oil resources can be effectively exploited [18]. Therefore, it is necessary to conduct a graded evaluation of shale oil resources to clarify the enriched resource distribution and guide shale oil exploration and development. Lu et al. indicated that the shale oil content (S_1 or “A”) showed “trichotomy” with the increase in TOC [19]. Specifically, when the TOC is large, S_1 is characterized by relatively stable large values, corresponding to the enriched resources or saturated resources; however, if TOC is lower, S_1 locates in the low value-region, associated with the dispersed or saturated resources; if TOC is between these two regions, S_1 increases significantly and is intermediate, which is called low-efficiency or undersaturated resources. However, the current shale oil resource classification criteria are mainly for residual shale hydrocarbons and resource classification evaluation criteria based on the total shale oil content have not been established.

The logging evaluation method has the advantages of a shorter cycle, higher resolution, and more visual and continuous characteristics. There is a certain response relationship between various logging parameters and TOC, S_1 , and S_2 . Therefore, the prediction model of TOC, S_1 , and S_2 quantitative prediction can be established. Domestic and foreign scholars have proposed a variety of methods to predict the TOC of hydrocarbon source rocks based on logging data, such as the multiple linear regression method, ΔLogR method, BP neural network method, etc. Different methods have different principles, advantages, and disadvantages. They are applicable to different geological conditions and data conditions.

The shale oil and gas resource evaluation methods mainly include analogous, statistical, genesis, comprehensive analysis, and volumetric techniques. Due to the low degree of shale oil exploration in China, the analogous method is unsuitable [20] because of the lack of connection with the actual geological conditions of oil and gas fields. The statistical approach is only suitable for evaluating and predicting oil and gas reservoirs or basin areas in the middle and late stages of exploration and development [21]. The genetic method cannot obtain the residual hydrocarbon occurrence mechanism and enrichment pattern and has parameter dependence in the calculation process, resulting in the accuracy of the calcu-

lation results being constrained [22]. Therefore, the genesis method may not be suitable for resource evaluation of shale reservoirs with low moveability. The integrated analysis method must be based on specific development history information within the exploration study area. This method is challenging to apply in a shale oil and gas exploration area lacking drilling and production data. The volumetric method divides the rocks into unit masses based on a spatial model of hydrocarbon source rocks for the target area of shale oil exploration. The oil content per unit mass is then used to quantitatively evaluate the shale oil resources of the entire hydrocarbon-bearing region [23].

In this paper, the corrected amount of heavy hydrocarbon was obtained by comparing the difference of S_2 before and after oil washing based on the room-temperature samples. The cores were treated combined with liquid nitrogen freezing to prevent light hydrocarbon loss. The correction amount of light hydrocarbon was determined by comparing it with the pyrolysis parameters of the room-temperature samples. Based on the characteristic of triple-division between the total oil content and TOC, the shale oil resource grading evaluation criteria in the study area were determined. In addition, the volumetric method was used to calculate the total enriched shale oil resources in the E_1f_2 of Gaoyou Sag. This study is helpful for the shale oil exploration and development in Gaoyou Sag, Subei Basin.

2. Geology Setting

The Subei Basin is the onshore part of the Subei South Yellow Sea Basin, which is bounded to the north and south by the South Jiangsu Uplift and the Lusu Ancient Land, respectively, west to the Tanlu Fault and east to the South Yellow Sea Basin, with an area of $3.8 \times 10^4 \text{ km}^2$ (Figure 1) [24]. The Subei Basin is divided into two depressions in the north and south by the Jianhu Uplift, namely the Dongtai and Yanfu Depressions. The Dongtai Depression is divided into six sags (Jinhu, Linze, Gaoyou, Baiju, Qintong, and Haian) and eight salients (Lintangqiao, Liubao, Tuoduo, Wubao, Yuhua, Xiaohai, Taizhou, and Liangduo). The Gaoyou Sag is located in the central part of the Dongtai Depression and the Cenozoic strata, such as the Taizhou (K_2t), Funing (E_1f), and Dainan (E_2d) Formations, are deposited from the bottom up. The shale in E_1f_2 is the most developed and the primary exploration area for shale oil in the Subei Basin [25].

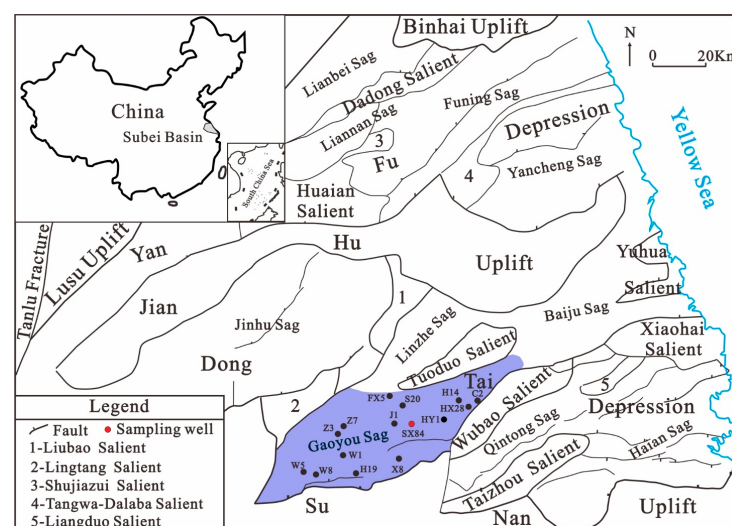


Figure 1. Geological setting of the study area [26].

3. Methodology

3.1. Samples and Experiments

In this study, a total of 119 shale samples were collected from Well SX84 in the Gaoyou Sag. Additionally, 92 samples were collected for the liquefrozen Rock-Eval measurements, which means that the sample pretreatments were conducted at the temperature of liquid nitrogen—at about -196°C . The TOC and Rock-Eval tests were carried out using the

Leco-SC 230 and Rock-Eval VI instruments. Furthermore, the Rock-Eval experiments were performed on the as-received and oil-washed shale samples. This study adopted the mixed solvent of dichloromethane and acetone (3:1 in volume) to eliminate the residual oil in shales under the temperature of 80 °C and the pressure of 0.2 MPa. These samples were then dried in a vacuum oven at 110 °C for 24 h.

3.2. Correction of Light and Heavy Hydrocarbon Loss for S_1

S_2 represents the generated hydrocarbons from kerogen when the temperature gradually rises from 300 °C to 600 °C during rock pyrolysis. However, some heavy hydrocarbons already present in the shale have boiling points much higher than 300 °C. Moreover, the adsorption and swelling of kerogen cause it to be impossible for the residual hydrocarbons to be completely volatilized before 300 °C, corresponding to this residual hydrocarbon being detected as S_2 . If there is no heavy hydrocarbon loss from the shale, S_2 (from room-temperature tests) the values are nearly equal to the S_{2L} (from liquefrozen tests). If the hydrocarbon in the room-temperature sample after being oil-washed is basically removed, the hydrocarbon in S_2 is all from kerogen pyrolysis. Therefore, the difference between S_2 and S_2' is the corrected amount of heavy hydrocarbon for S_1 . The corrected amount of heavy hydrocarbon can be written as follows:

$$\Delta S_2 = k_2 \cdot S_2, \quad (1)$$

where k_2 is the heavy hydrocarbon correction index.

S_1 refers to the volatilized hydrocarbons during the rock pyrolysis analysis when temperatures are less than 300 °C, which are C_{7-33} hydrocarbons. However, shale light hydrocarbons (C_{6-13}) have primarily dissipated due to the long placement time. The liquefrozen technology is the most effective and straightforward method to correct light hydrocarbons [26]. Since the shale samples were cored on site and then processed at liquid nitrogen temperatures, the light hydrocarbon losses are negligible (the temperature of liquid nitrogen is about −196 °C and the melting point of methane is about −182.6 °C). Subsequently, the S_{1L} was measured from the liquefrozen shale. The light hydrocarbon correction equation can be obtained by establishing a linear relationship between S_1 and S_{1L} . Therefore, the S_1 light hydrocarbon correction model can be determined as follows:

$$S_{1L} = k_1 \cdot S_1, \quad (2)$$

where k_1 is the light hydrocarbon correction factor. Then, the total shale oil content (S_{1O}) can be obtained.

$$S_{1O} = k_1 \cdot S_1 + k_2 \cdot S_2. \quad (3)$$

3.3. $\Delta \log R$ Method

The logging evaluation method has the advantages of a shorter cycle, higher resolution, and more visual and continuous characteristics [27]. The $\Delta \log R$ method was widely used to estimate organic matter (TOC) or oil (S_1 or “A”) contents in shales based on the acoustic interval transit time (AC) and resistivity (Rt) logging curves. However, using a fixed empirical formula or coefficient to estimate the TOC of source rocks is not objective because the properties and well-logging response characteristics of source rocks in different areas are obviously different. Therefore, these methods have been improved in three aspects: the value of the superposition coefficient, the determination of the baseline value, and the control of the influence of maturity. The improved superposition coefficient is determined based on the log data in the study area and the baseline value is automatically identified. [28,29]. The model equation is as follows:

$$\Delta \log R = \lg R + \lg(R_{\max}/R_{\min})/(\Delta t_{\max} - \Delta t_{\min}) \cdot (\Delta t - \Delta t_{\max}) - \lg R_{\min} \quad (4)$$

$$TOC = a \cdot \Delta \log R + b, \quad (5)$$

where R is the value of the resistivity curve, $\Omega \cdot m$; Δt is the value of acoustic time difference, $\mu s/ft$; $R_{min/max}$ is the maximum/minimum value of resistivity, $\Omega \cdot m$; $\Delta t_{min/max}$ is the maximum/minimum value of acoustic time difference, $\mu s/ft$; and a and b are the coefficients in the fitted equation, corresponding to the maturity parameter and background value of TOC content, respectively. The organic matter of the E_1f_2 in the study area has high heterogeneity. Therefore, the improved $\Delta \log R$ method was used in this study to estimate the TOC , S_1 , and S_2 of the shale.

4. Results and Discussion

4.1. Shale Organic Geochemical Characteristics

The TOC contents of the selected shales range from 0.19% to 3.4%, with a mean of 1.02%, mainly in the range of 0–2% (Figure 2). The S_1 is between 0.03 mg/g and 2.67 mg/g (mean 0.76 mg/g) and is primarily distributed in a range of 0–1.5 mg/g (Figure 3a). The S_2 ranges from 0.15 to 4.9 mg/g, with an average of 1.87 mg/g, mainly in the 1–4 mg/g (Figure 3b).

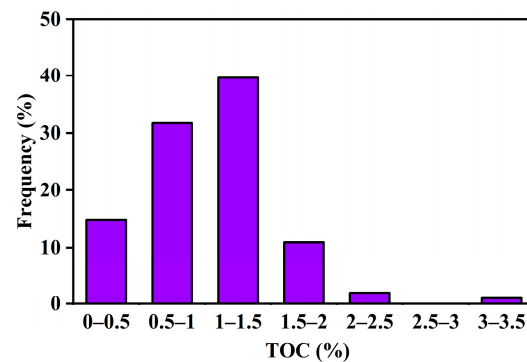


Figure 2. Distribution of TOC content.

The $S_{1'}$, obtained from the oil-washing samples, range from 0.08 mg/g to 0.19 mg/g, with an average of 0.13 mg/g, which is significantly lower than S_1 (Figure 3a,e). The results mean that the residual oil was removed by the oil washing. The $S_{2'}$ (related to oil-washing shales) varies from 0.24 mg/g to 0.81 mg/g, with an average of 0.59 mg/g, which is lower than S_2 (Figure 3b,f), implying that there is a part of heavy hydrocarbons that could not be detected in S_1 but calculated to S_2 . The S_{1L} associated with liquefrozen shale varies from 0.05 mg/g to 4.64 mg/g (mean 1.22 mg/g) and is primarily distributed in a range of 0–3 mg/g (Figure 3c). S_{1L} is greater than S_1 , indicating the light-medium hydrocarbon loss in the room-temperature sample. The corresponding S_{2L} is between 0.21 and 5.24 mg/g, mainly in the 1–4 mg/g range, with an average of 2.23 mg/g (Figure 3d).

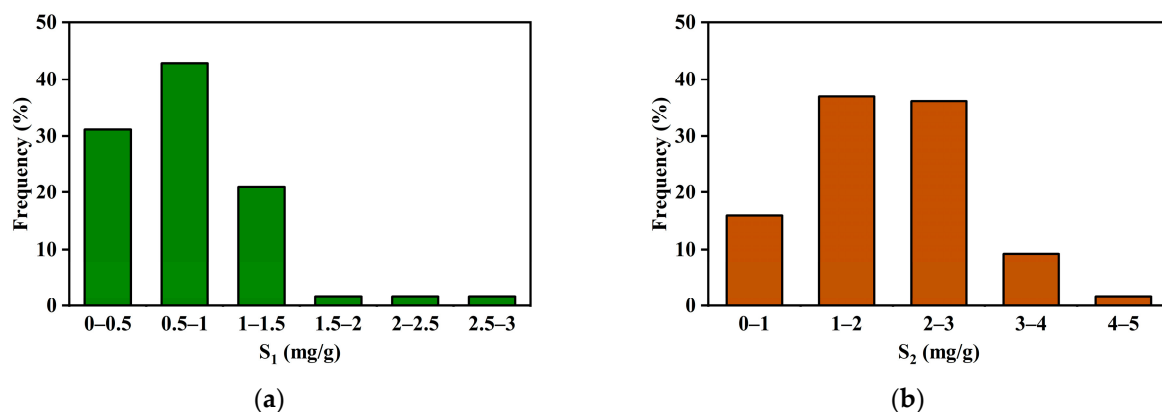


Figure 3. Cont.

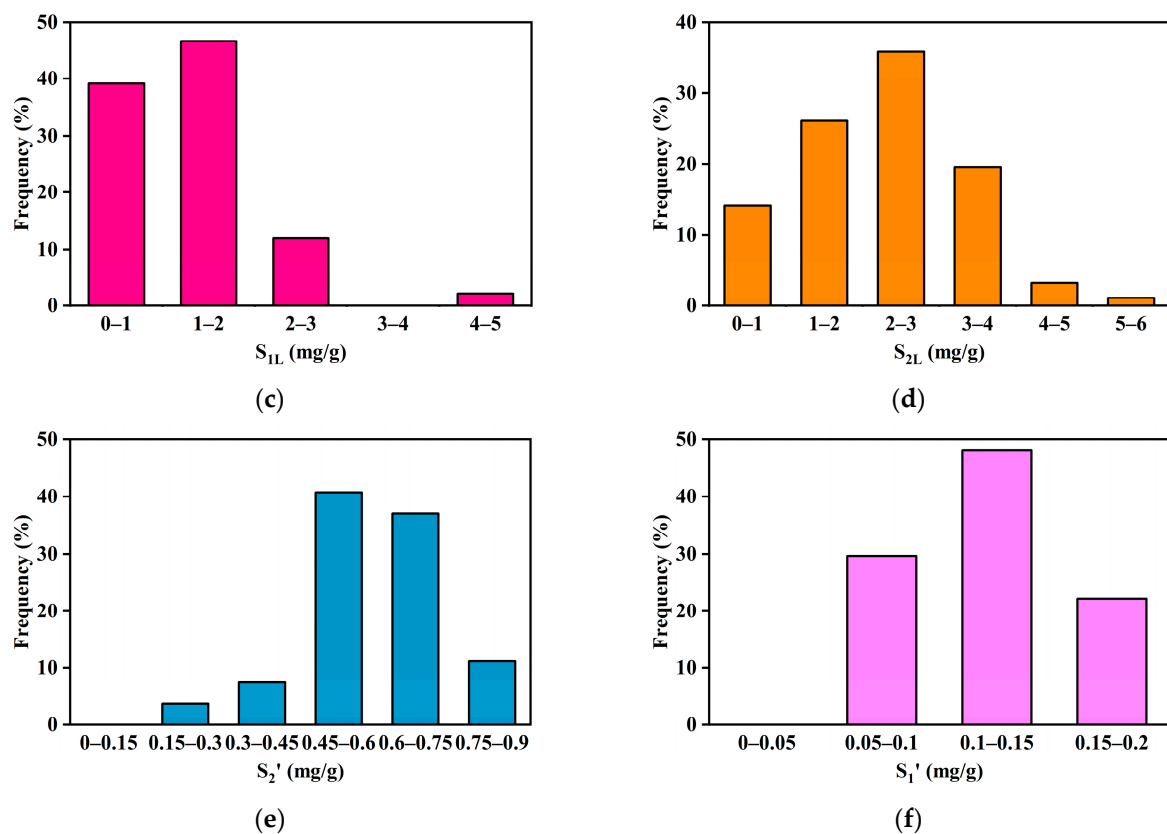


Figure 3. Rock-Eval parameters obtained from the liquefrozen, as-received, and oil-washing shales. (a) Distribution of S_1 content. (b) Distribution of S_2 content. (c) Distribution of S_{1L} content. (d) Distribution of S_{2L} content. (e) Distribution of $S_{2'}$ content. (f) Distribution of $S_{1'}$ content.

4.2. Correction Model of Heavy and Light Hydrocarbon Loss for S_1

As illustrated in Figure 4a, values of $S_{1'}$ are commonly lower than those of S_1 , indicating that the residual oil was almost completely removed. However, S_2 agrees well with the S_{2L} , with a large correlation coefficient ($R^2 = 0.82$) (Figure 4b), implying that almost no heavy hydrocarbons were lost. Therefore, the room-temperature shales can be used to determine the heavy hydrocarbon correction model. The difference (ΔS_2) between S_2 and $S_{2'}$ correlates well with S_2 with a slope of 0.69 ($R^2 = 0.9$), as exhibited in Figure 5a. Thus, the S_1 heavy hydrocarbon correction amount can be determined as about $0.69 \cdot S_2$.

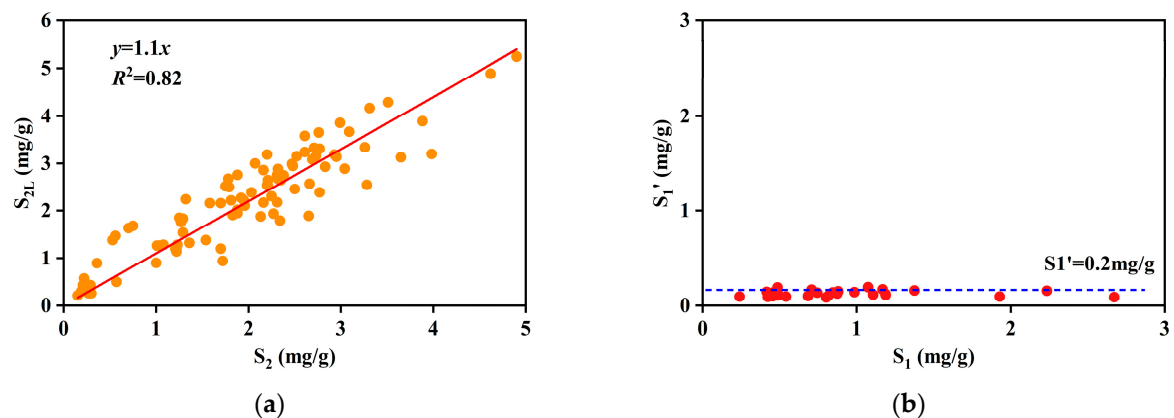


Figure 4. Relationships between room-temperature and liquefrozen shales values of S_2 (a) and relationships between before and after oil washing values of S_1 (b).

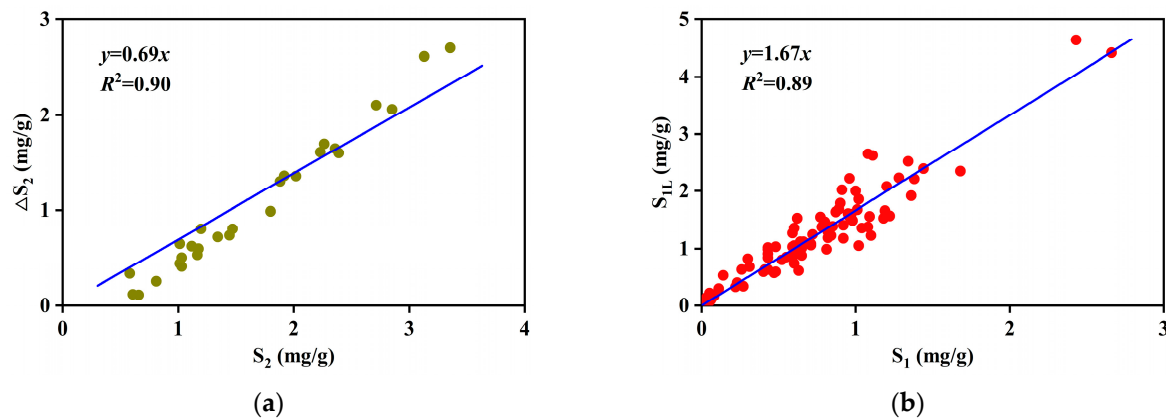


Figure 5. Heavy (a) and light (b) hydrocarbon correction models.

Figure 5b shows that S_{1L} obtained from the liquefrozen shales correlates linearly with S_1 received from the room-temperature shales, with a slope of 1.67. Thus, the shale S_1 light hydrocarbon correction factor is 1.67 and the light hydrocarbon compensation is $0.67 \cdot S_1$. Additionally, the total shale oil content (S_{1O}) is:

$$S_{1O} = 1.67 \cdot S_1 + 0.69 \cdot S_2. \quad (6)$$

4.3. Classification of Shale Oil Resource

According to the heavy and light hydrocarbon correction models, the total oil contents in shales were determined. The total shale oil contents range from 0.15 mg/g to 7.63 mg/g (mean 2.55 mg/g), which is about 3.2 times that of tested S_1 .

As displayed in Figure 6, the shale oil resource classification can be established according to the “trichotomy” between TOC and S_{1O} . When the TOC is low ($<0.6\%$), S_{1O} shows a lower value (<1.8 mg/g), corresponding to the ineffective resource. When the TOC increases from 0.6% to 1.5%, S_{1O} gradually increases, corresponding to the low-efficiency resources. However, if the TOC is larger than 1.5%, S_{1O} is characterized by a high value corresponding to the enriched resource. Therefore, the shale oil resource classification of the E_1f_2 in the Gaoyou Sag was evaluated as follows: enriched resource ($TOC \geq 1.5\%$, $S_{1O} \geq 4.0$ mg/g), low-efficiency resource ($0.6\% \leq TOC < 1.5\%$, 1.8 mg/g $\leq S_{1O} < 4.0$ mg/g), and ineffective resource ($TOC < 0.6\%$, $S_{1O} < 1.8$ mg/g) (Figure 6).

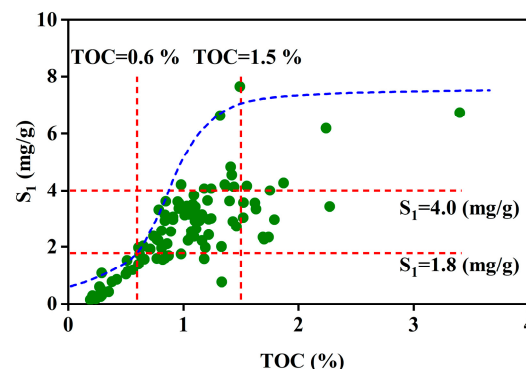


Figure 6. Relationship between TOC and S_{1O} .

According to the resource classification criteria established above, the main type of shale oil in the Gaoyou Sag is inefficient resources.

4.4. Estimation of Shale Oil Resource

4.4.1. Calculations of TOC , S_1 , and S_2 by $\Delta\log R$

The improved $\Delta\log R$ model was used to estimate the TOC , S_1 , and S_2 of the E_1f_2 and the estimated models were determined based on the tested values from the Well SX84 located in the Gaoyou Sag. The reliability of the logging data of the shale section with a thickness of less than 1 m was carefully studied. As exhibited in Figure 7a, the TOC shows a significant positive correlation with amplitude difference ($R^2 = 0.85$). The TOC can be calculated as follows:

$$TOC = 0.62 \cdot \Delta\log R - 0.27. \quad (7)$$

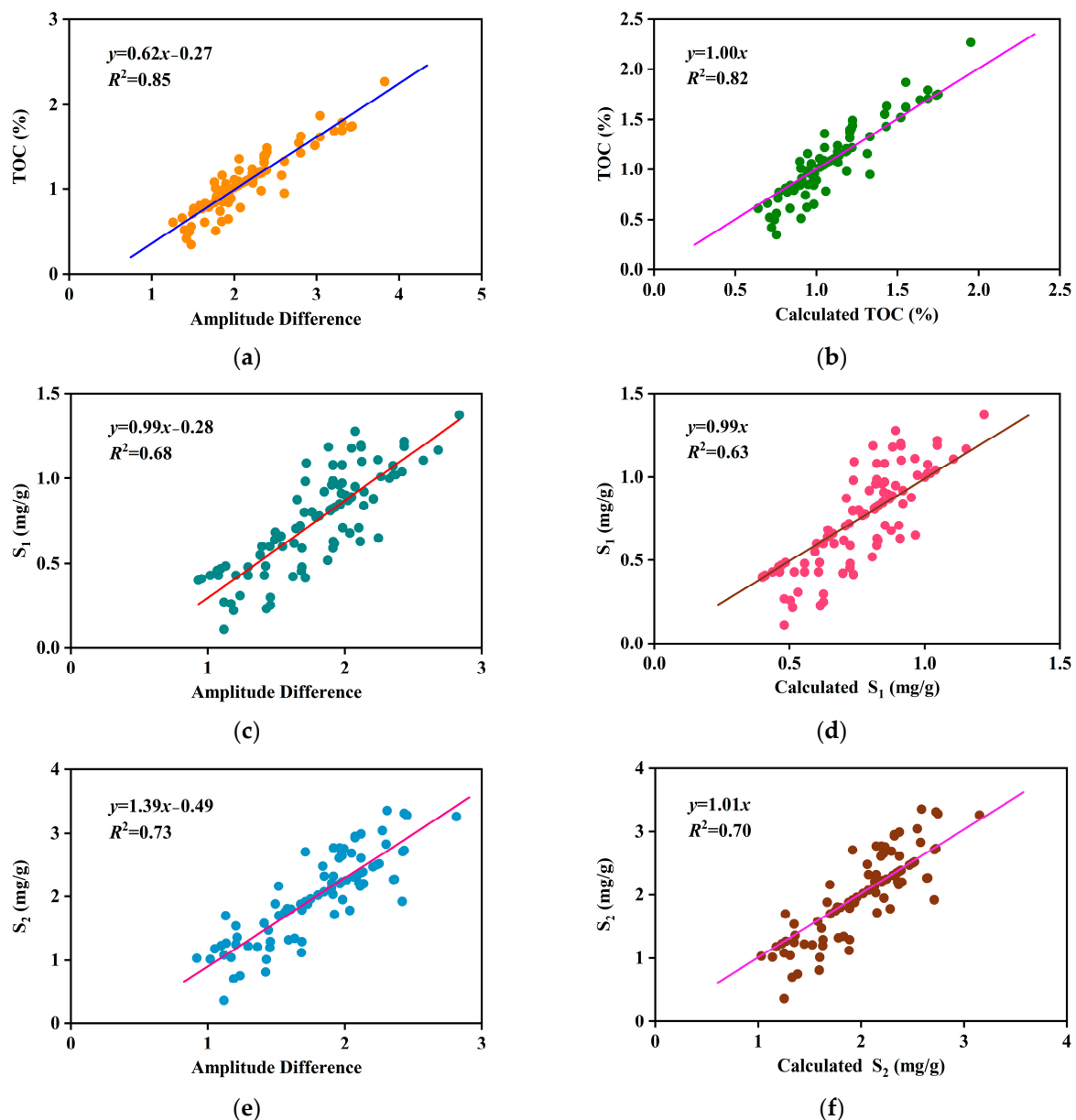


Figure 7. Relationships between calculated and measured TOC , S_1 , and S_2 (b,d,f). Relationship between amplitude difference and TOC , S_1 , and S_2 (a,c,e).

The estimated TOC agrees well with the tested TOC ($R^2 = 0.82$), indicating that the calculation values are reliable. The estimated models of S_1 and S_2 were obtained using the same method. Additionally, the calculated S_1 and S_2 values show good positive correlations with magnitude differences. Moreover, the calculated S_1 values correspond

well with tested S_1 and so do the calculated and tested S_2 , as displayed in Figure 7d,f. The S_1 and S_2 estimated models are as follows.

$$S_1 = 0.99 \cdot \Delta \log R - 0.28. \quad (8)$$

$$S_2 = 1.39 \cdot \Delta \log R - 0.49. \quad (9)$$

The established models were applied to estimate the TOC, S_1 , and S_2 of the E_1f_2 in the Gaoyou Sag. In this study, a total of 17 wells were assessed (Figure 1) and the Wells SX84 and HY1 are shown in Figures 8 and 9. The total shale oil contents of Well HY1 are mainly larger than those of Well SX84. The total shale oil contents of Well HY1 range from 2.32 mg/g to 12.78 mg/g (mean 5.39 mg/g), while the average total shale oil contents in the Well SX84 is 2.46 mg/g (1.12–8.30 mg/g). More resource-rich shale oil occurs in Well HY1.

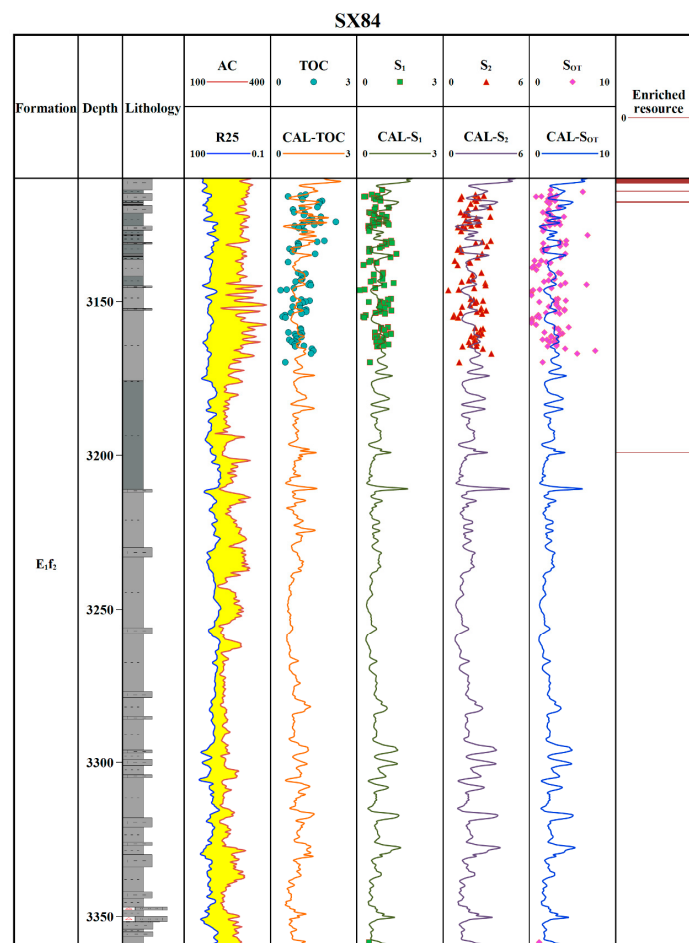


Figure 8. Results calculated using the $\Delta \log R$ method of Well SX84.

4.4.2. Shale Oil Resource in Gaoyou Sag

The total shale oil and enriched resources were calculated using the volumetric method based on the recovered S_1 of the E_1f_2 in the Gaoyou Sag. The volumetric method of calculating shale oil resources is provided by the following formula:

$$Q = \sum 10^{-1} \times S \times H \times \rho \times S_1, \quad (10)$$

where Q is the single-well shale oil resource, 10^4 t/km²; S is the shale area, km²; H is the shale thickness, m; and ρ is the shale density, g/cm³. The calculation results show that the total shale oil resources in the study area are about 5.72×10^8 t, of which the enriched resource is about 1.70×10^8 t. According to the contour map of shale oil resources in the

Gaoyou Sag (Figure 10), enriched resource areas are mainly in the deep sag, especially near Well HX28.

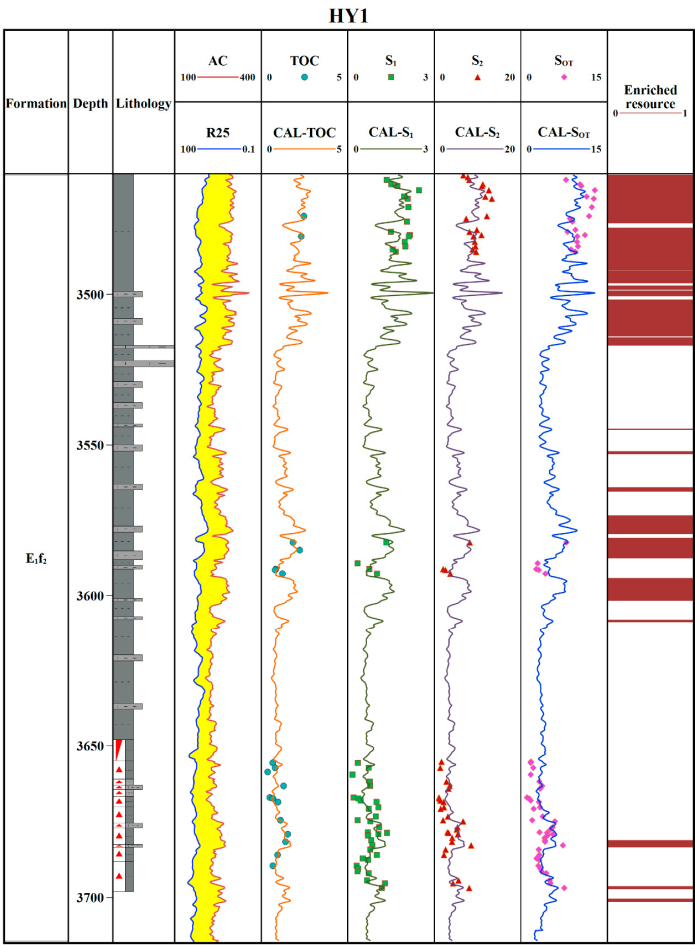


Figure 9. Results calculated using the $\Delta\log R$ method of Well HY1. (The red triangles in Lithologic series mean oil immersion).

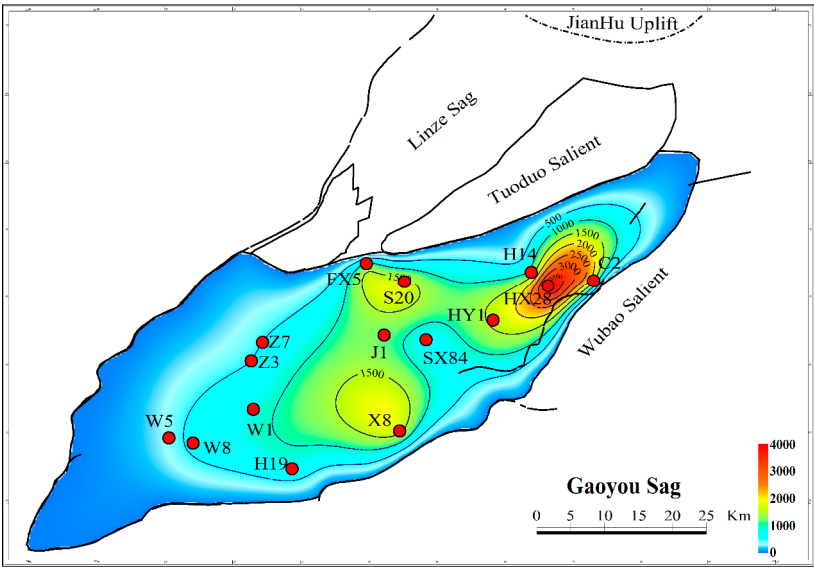


Figure 10. Contour map of the oil resource in Gaoyou Sag.

5. Conclusions

The light and heavy hydrocarbon correction models of S_1 were developed based on the rock pyrolysis of liquefrozen, conventional, and oil-washed shales. The shale S_1 heavy hydrocarbon correction content is about $0.69 \cdot S_2$ and the light hydrocarbon compensation is $0.67 \cdot S_1$. The corrected S_1 is about 3.2 times more than the conventional shale-tested value.

The shale oil enriched resource classification of the E_1f_2 in the Gaoyou Sag was $TOC \geq 1.5\%$ and $S_{10} \geq 4.0$ mg/g, the low-efficiency resource was $0.6\% \leq TOC < 1.5\%$ and 1.8 mg/g $\leq S_{10} < 4.0$ mg/g, and the ineffective resource was $TOC < 0.6\%$ and $S_{10} < 1.8$ mg/g.

The total shale oil and enriched resources were calculated using the volumetric method. The total shale oil resource in the Gaoyou Sag is about 5.72×10^8 t, of which the enriched resource is about 1.70×10^8 t. The enrichment areas are mainly in the deep sag, especially near Well HX28.

Author Contributions: Conceptualization, Q.Z. and S.L.; methodology, Q.Z.; software, Q.Z.; validation, Q.Z., P.Z. and H.H.; formal analysis, Q.Z.; investigation, J.W.; resources, J.W.; data curation, Z.L.; writing—original draft preparation, Q.Z.; writing—review and editing, Q.Z.; visualization, Q.Z.; supervision, P.Z.; project administration, S.L.; funding acquisition, S.L. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

Nomenclature

S_1	residual hydrocarbon obtained from room-temperature shale using rock pyrolysis, mg/g;
S_2	hydrocarbon from kerogen pyrolysis obtained from room-temperature shale using rock pyrolysis, mg/g;
S_{1L}	residual hydrocarbon obtained from liquefrozen shale using rock pyrolysis, mg/g;
S_{2L}	hydrocarbon from kerogen pyrolysis obtained from liquefrozen shale using rock pyrolysis, mg/g;
S_1'	residual hydrocarbon obtained from room-temperature shale after oil-washed using rock pyrolysis, mg/g;
S_2'	hydrocarbon from kerogen pyrolysis obtained from oil-washed shale using rock pyrolysis, mg/g;
ΔS_2	difference between S_2 and S_2' , mg/g;
S_{10}	total oil, mg/g.

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