

Exploring the influence of heating rate on the permeability of Fushun oil shale

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Abstract. Oil shale from the Fushun West Open-pit Mine was selected as the research object to study the influence of heating rate on permeability. Through pyrolysis experiments at different heating rates, the evolution of permeability under various heating conditions was explored. Response surface methodology was used to optimize the pyrolysis parameters, and the optimal parameter combination was determined: a heating rate of 34.55 °C/min, a final reaction temperature of 475 °C, and a constant-temperature holding duration of 131 min. Under these conditions, the pyrolysis efficiency of Fushun oil shale was optimal. The key parameters identified provide a valuable reference for the in-situ exploitation of Fushun oil shale.

Keywords: oil shale, heating rate, permeability, response surface optimization.

1. Introduction

The 2025 international Unconventional Resources Technology Conference (URTeC) highlights that unconventional resources, particularly oil shale, provide lasting solutions to global energy challenges. Green and high-value utilization has become the core orientation [1].

In terms of advanced mining technologies, Pan et al. [2] and Li et al. [3] summarized sophisticated approaches such as the in-situ conversion process (ICP), geothermal fuel cell (GFC) technology, supercritical water heating (SWH), and voltage-induced fracturing. Crucially, Liu et al. [4] proved that

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these technologies drive pore network evolution through fundamentally distinct mechanisms. For conductive heating, Zhao et al. [5] experimentally confirmed that ICP's slow heating triggers massive mineral decomposition at 350 °C, increasing porosity up to tenfold. Similarly, Luo et al. [6] found that GFC's steady mid-temperature heat transforms isolated pores into a highly connected bimodal network with a peak pore size of 15.2 nm. Conversely, for convective heating, Kang et al. [7] reported that SWH utilizes hydrothermal effects to extract pore-blocking bitumen and catalyze clay illitization, generating fractures up to 150 μm wide – five times larger than those generated by dry pyrolysis. Finally, Bauer et al. [8] demonstrated that voltage-induced fracturing uses electromechanical shockwaves to physically shatter the rock matrix, creating macro-scale channels that resist closure under a 58.6 MPa hydrostatic pressure and increase permeability by up to nine orders of magnitude. Consequently, optimizing fluid migration requires quantifying how specific heating rates and thermodynamic parameters drive this microscopic structural evolution under each distinct technology.

In terms of experiments and simulations, although oil shale holds great promise, research on how heating rates impact its macroscopic permeability remains insufficient. The permeability enhancement generally transitions from a pore-structure-dominated regime at low temperatures to a fracture-dominated regime at high temperatures [9–10]. Schrodtt et al. [11] studied the effect of the heating rate on the pore structure of U.S. oil shale using nitrogen and carbon dioxide as pyrolysis atmospheres. The results indicated that during the low-temperature stage of the reaction (< 300 °C), hot gas products precipitated to a certain extent to fill the pore volume, and the pore area decreased. At high temperatures (300–600 °C), the pore area increased significantly, while the pore surface area changed little at different heating rates. Sun et al. [12] analyzed the evolution of pores and fractures in the pyrolysis solids of oil shale using nitrogen isothermal adsorption experiments. With increasing heating rate, micropores and mesopores formed fractures that developed into fracture networks. Li et al. [13] and Yu et al. [14] simulated in-situ extraction conditions, demonstrating that optimizing the heating temperature is vital for hydrocarbon generation and fracture propagation. Recently, increasing attention has been paid to Fushun oil shale to investigate its thermal cracking characteristics and dynamic permeability evolution [7].

Liu et al. [4] comprehensively reviewed the thermal cracking process, pore evolution, and permeability enhancement of oil shale under various in-situ heating conditions. To further observe the internal structural changes, Zhang et al. [15] utilized advanced computed tomography imaging techniques to characterize how temperature and heating media influence the mesostructural evolution of fractured shale during in-situ pyrolysis. Additionally, Abdi-Khanghah et al. [16] employed non-isothermal kinetic modeling to optimize the thermodynamic parameters for maximizing the hydrocarbon yield of shale reservoirs. However, as pointed out by Yang et al. [17], while the fundamental mechanisms of pyrolysis and fracture generation have been

widely documented, comprehensive studies quantitatively evaluating the coupled effects of different heating rates on macroscopic permeability remain limited [18].

In view of the above factors, relevant experimental studies on the influence of heating rates, as well as parameter optimization based on response surface methodology (RSM), are currently lacking. Therefore, it is necessary to design pyrolysis experiments using Fushun oil shale to obtain the variation in permeability under different heating rates. By applying RSM to establish a predictive quadratic regression model, we aim to determine the optimal parameter combinations required to maximize fluid migration capacity. This study provides a reference for investigating temperature-dependent behavior in the field of in-situ oil shale extraction.

2. Experimental study on the permeability of Fushun oil shale at different heating rates

2.1. Experimental materials, instruments, and samples

Oil shale from the Fushun West Open-pit Mine was used as the experimental material, and mixed oil shale samples extracted from each quarter were used. Fushun oil shale originates from the Fushun area of Liaoning Province in northern China. The mineral composition includes kaolinite, montmorillonite, illite, calcite, pyrite, etc., and the surface is rich in bituminous luster. Fushun oil shale has a high organic carbon content, with a maximum oil content of up to 12% and an average oil content of around 5% to 8%. Fushun oil shale is rich in Al_2O_3 and SiO_2 and has a high melting point.

Experimental measurements of oil shale permeability at different heating rates were conducted, and the instruments and equipment used in the experiments are shown in Table 1.

Table 1. Experimental equipment

Device	Equipment model	Manufacturer
Digital display forced air drying oven	GZX-9023 MBE	Shanghai Boxun Industrial Medical Company
Electronic analytical balance	BSA223S+	Beijing Sartorius Instruments Co., Ltd.
Automated dust-free core preparation equipment	KDZQGM-II	Jiangsu Kedi Petroleum Instrument Co., Ltd.
Permeability meter	STY-II gas permeability meter	Nantong Huaxing Petroleum Instrument Co., Ltd.
Muffle furnace	SXL-1008 Programmable box-type electric furnace	Shanghai Jinghong Laboratory Instrument Co., Ltd.

Before the experiment, the samples were processed. The samples were cut into cylindrical cores with a diameter of 50 mm and a length of 20 to 70 mm using a drilling machine. A total of 18 sets of experiments were conducted. The oil shale cores were then dried in an oven at 105 °C for 24 hours and subsequently placed in a desiccator to cool to room temperature.

2.2. Experimental methodology

2.2.1. Experimental conditions

Oil shale core samples were pyrolyzed at different heating rates, and the permeability of the cores was measured at each temperature point. The heating rates were set at 10, 20, 30, 40, 50, and 60 °C/min, and the pyrolysis temperature points were set at 300, 400, 500, and 600 °C.

2.2.2. Preparation of the permeability instrument

- Measure the core sample length and diameter with a vernier caliper and calculate the cross-sectional area A .
- Insert the standard core block into the core holder and turn the hand wheel upward until the top of the sample fits tightly against the top of the holder and is securely fixed.
- Connect the nitrogen cylinder. Open the gas source valve (the gas source pressure is pre-adjusted) and open the ring pressure valve (the ring pressure is pre-adjusted). After the pressure on the ring pressure gauge stabilizes, close the ring pressure valve.
- Adjust the pressure-regulating valve. When the pressure gauge reaches the pressure required for the experiment, open the measuring valve and read the float flowmeter flow at different pressures.
- Adjust the pressure-regulating valve to zero pressure, vent through the ring pressure valve to zero ring pressure, and then repeat the above steps to remove the rock sample.

A diagram of the core permeability measurement system is shown in Figure 1.

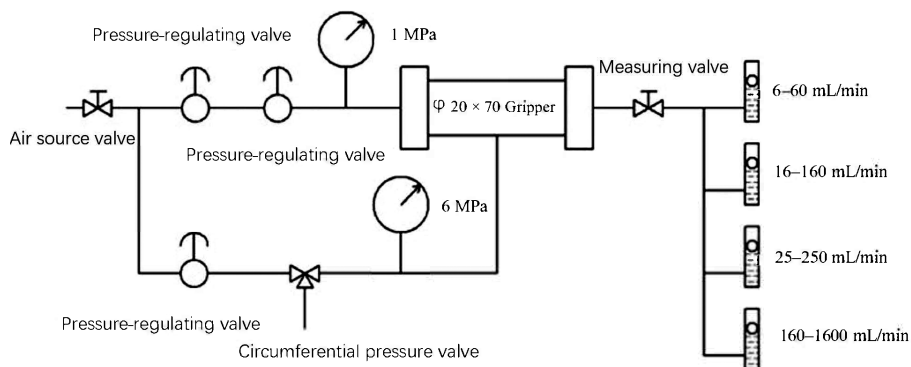


Fig. 1. Schematic diagram of the permeability measurement system.

2.2.3. Experimental procedure

- Record the core sample dimensions (diameter D , length L , and cross-sectional area A) and the experimental conditions (gas viscosity μ , chamber temperature t , and atmospheric pressure P_a). Permeability was calculated from the measured pressure drop and flow rate using Darcy's law.
- Since actual measurements are influenced by pressure and temperature variations, and given the high compressibility and thermal expansion of gases in the measurement environment, flow rate correction is necessary. Specifically, one of the four miniature float flowmeters (with different measurement ranges) equipped with the gas permeability meter should be selected during measurements. Where conditions permit, the flowmeter with the smallest applicable range is preferred. The calculation formula is as follows:

$$Q_0 = 0.2853Q'_0 \sqrt{\frac{1}{p_a(273 + t) \times 10}}, \quad (1)$$

where Q_0 is the corrected actual flow rate, Q'_0 is the flowmeter reading, P_a is the absolute atmospheric pressure, and t is the room temperature.

- Record the pressures P_1 and P_2 at both ends of the core as gas flows through it, then calculate the average pressure ($\bar{P} = (P_1 + P_2)/2$) and record the gas volumetric flow rate ($\bar{Q}$). Finally, substitute these parameters into Darcy's formula.

The percolation law of a unidirectional fluid through the core only conforms to Darcy's law when the pressure gradient is small and the flow rate is low. When the pressure gradient exceeds this limit, it no longer follows Darcy's law but instead follows a nonlinear percolation law. The results are described by Equation 2:

$$k_g = \frac{2Q_2L\mu p_0}{A(p_1^2 - p_2^2)} \times 10^{-1}, \quad (2)$$

where k_g is the gas permeability, Q_2 is the gas flow rate at the core outlet end, L is the core length, μ is the gas viscosity at test temperature and atmospheric pressure, P_0 is the atmospheric pressure, A is the cross-sectional area of the core, P_1 is the absolute pressure at the core inlet end, and P_2 is the absolute pressure at the core outlet end.

2.3. Results of the oil shale permeability test

The pyrolysis environment was monitored by recording core pressures and flow rates, with permeability calculated using Darcy's law (Fig. 2). Permeability exhibits a phased growth trajectory as temperature increases.

Phase I: Pore-structure-dominated evolution (20–300 °C): Permeability remains extremely low. Minor fluctuations driven by water evaporation and thermal expansion create isolated micro-pores that lack the connectivity needed for effective fluid migration.

Phase II: Fracture-dominated evolution (300–500 °C): An exponential surge in permeability occurs. Above 380 °C, intense kerogen pyrolysis generates massive amounts of volatile hydrocarbons, causing localized fluid overpressure that forcefully drives rapid micro-fracture propagation.

Phase III: Mineral-transformation-supported evolution (500–600 °C): With organic pyrolysis nearly complete, explosive permeability growth decelerates. Further minor enhancements are sustained primarily by mineral phase transformations.

In summary, the macroscopic permeability evolution of Fushun oil shale is fundamentally fracture-dominated. Organic matter pyrolysis is the absolute dominant driver, creating severe fluid overpressure that shatters the rock matrix to exponentially increase permeability. Conversely, mineral transformations play only a secondary, auxiliary role, sustaining slow permeability increases at extremely high temperatures (500–600 °C).

The change in permeability is mainly due to the uneven thermal expansion of special mineral matrices and high-temperature thermal reactions, such as the dehydroxylation of kaolinite at around 550 °C and of illite at around 580 °C. With the extension of pyrolysis time, the temperature of the oil shale core becomes sufficient to meet the pyrolysis conditions, and the uneven thermal stress within the core also causes fractures to expand and penetrate. As a result, the internal damage to the oil shale increases, and thermal fractures develop along the bedding direction. All these factors combined cause the permeability to increase slowly.

Regarding the effect of the heating rate on permeability, the experimental data reveal a non-linear evolutionary trend. In the active pyrolysis zone (400–500 °C), Yang et al. [17] demonstrated that higher heating rates induce more severe thermal shock and elevated temperature gradients, generating higher localized fluid overpressures that forcefully accelerate micro-fracture propagation. However, as the temperature approaches 600 °C, the correlation between heating rate and permeability enhancement shows no obvious trend. This plateau occurs because the primary organic matter has been completely exhausted, and permeability is now governed by competing secondary geomechanical mechanisms rather than volatile gas expansion. For instance, Li et al. [3] indicated that high temperatures and secondary reactions within the pores can lead to coke formation, which risks blocking narrow seepage channels. Furthermore, Zhao and Kang [19] experimentally confirmed that the extensive generation of fractures during the main pyrolysis phase severely compromises the compressive strength of the inorganic mineral skeleton, causing some unproped micro-fractures and pores to collapse or close under stress. Consequently, the ultimate permeability at 600 °C is dictated by the maximum structural damage sustained during the 400–500 °C phase and subsequent high-temperature mineral softening, rather than by the ongoing heating rate.

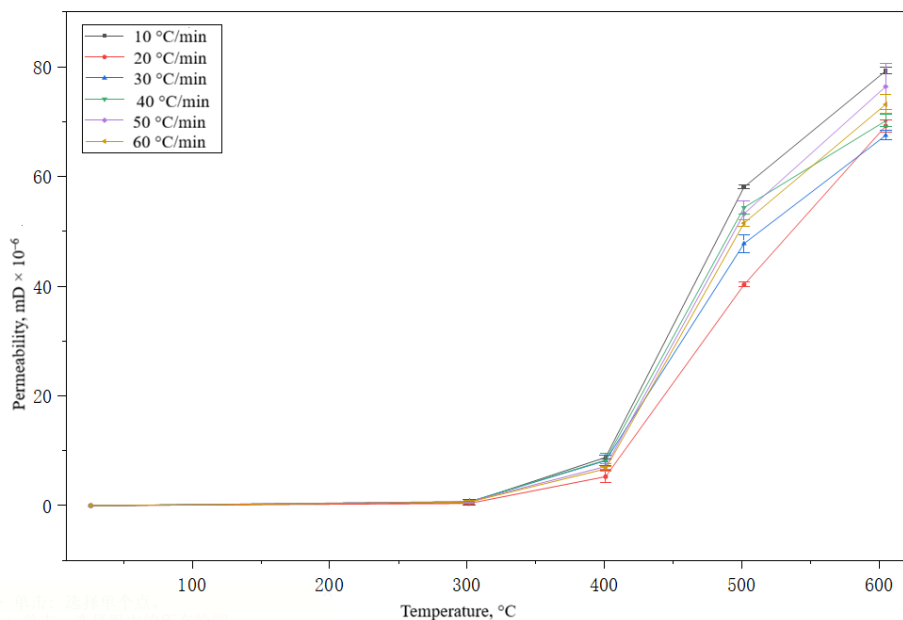


Fig. 2. Permeability curves of oil shale varying with temperature at different heating rates.

The oil shale samples are uniform and dense in their natural state before pyrolysis. With the increase in temperature, the decomposition of organic matter and the precipitation of inorganic minerals, the oil shale is more likely to develop cracks in the horizontal layer and connect and expand, resulting in significant changes in sample appearance and color. As the temperature reaches 300 °C, effective oil and gas seepage channels are formed along the horizontal bedding direction. The morphological changes of the core at temperatures of 20, 300, 400, 500, and 600 °C, including prominent features such as fracture propagation and structural fragmentation throughout the pyrolysis process, are presented in Figures 3 and 4.

The entire process shows a progressive trend of microfracture formation → local expansion → multidirectional development → local fracture → fracture expansion → reticular fracture → deep extension → complete dissociation, with fractures gradually evolving from surface damage to penetrating failure, accompanied by changes in the core structure from an intact state to complete fragmentation.

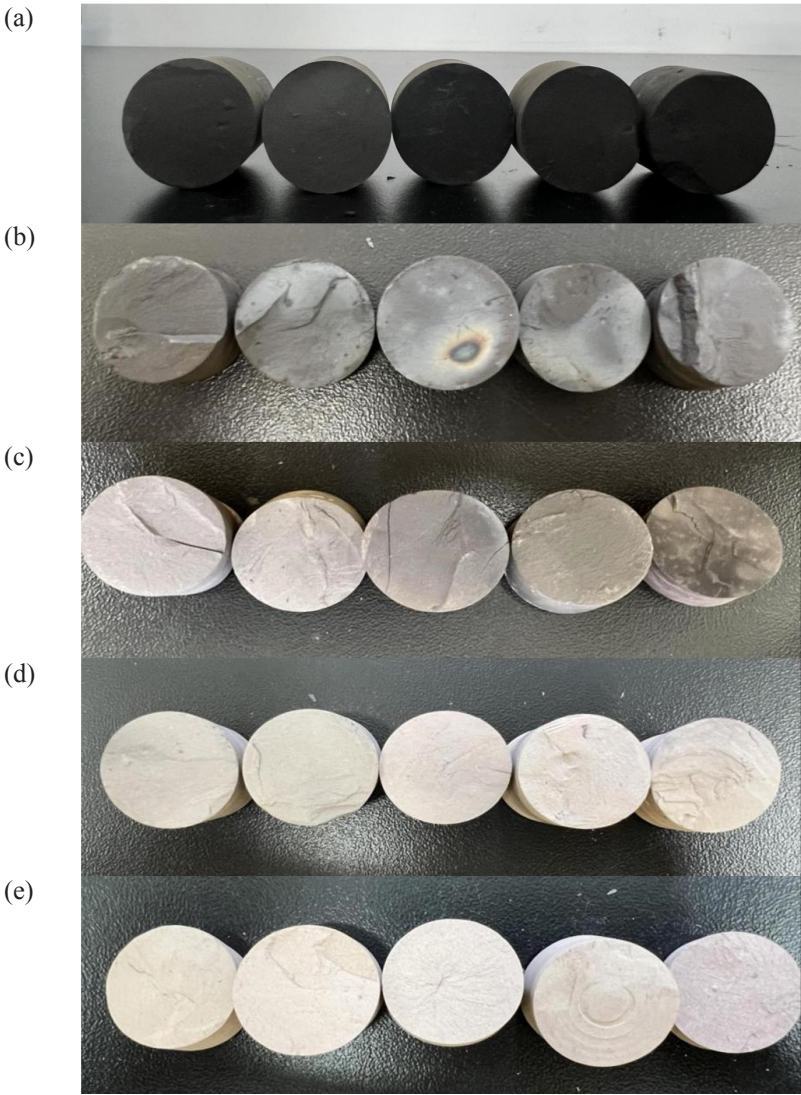


Fig. 3. Comparison of core sample states at different temperatures: (a) 20 °C, (b) 300 °C, (c) 400 °C, (d) 500 °C, and (e) 600 °C.

Note: To indicate the spatial scale, the standardized diameter of all cylindrical core samples shown in the images is exactly 50 mm, which serves as an internal scale reference. The lengths range from 20 mm to 70 mm.



Fig. 4. Core fracture propagation networks and structural failure conditions highlighting: (a) horizontal bedding expansion, and (b) vertical bedding fracture intersections.

Note: To indicate the spatial scale, the standardized diameter of all cylindrical core samples shown in the images is exactly 50 mm, which serves as an internal scale reference. The lengths range from 20 mm to 70 mm.

3. Analysis of pyrolysis data based on the response surface

3.1. Response variable – permeability optimization design

The pyrolysis experiments were conducted using multiple heating rates to test the permeability of the oil shale and optimize the response surface design. The heating rates were set at 10, 20, 30, 40, 50, and 60 °C/min to control the ambient temperature changes, and the final pyrolysis temperatures were set at 300, 400, 500, and 600 °C. After reaching the final pyrolysis temperature, the samples were maintained at a constant temperature for 0.5, 1, 2, and 3 h, respectively, followed by an optimization analysis of the permeability.

The optimized design approach was as follows. An experimental design was developed to optimize the permeability response surface with regard to the three response variables: heating rate, final pyrolysis temperature, and constant-temperature duration. A mathematical model was established using the Box–Behnken center combinatorial experimental design for response surface analysis. The quadratic multiple regression equation for oil shale pyrolysis permeability (Y_3) as a function of the independent variables A, B, and C is given in Equation 3.

To ensure absolute analytical clarity and mathematical consistency, the three critical independent variables are explicitly defined as follows:

Variable A: heating rate (°C/min)

Variable B: final pyrolysis temperature (°C)

Variable C: constant-temperature duration (h)

$$Y_3 = 1.345 \times 10^{-5} + 7.916 \times 10^{-7} A + 3.512 \times 10^{-5} B - 5.33 \times 10^{-6} C + 3.290 \times 10^{-6} AB + 2.347 \times 10^{-5} AC - 4.608 \times 10^{-6} BC - 1.171 \times 10^{-5} A^2 + 2.525 \times 10^{-5} B^2 - 8.708 \times 10^{-6} C^2 \quad (3)$$

The significant lack of fit (LOF) in Table 2 is a recognized statistical artifact known as the low pure-error phenomenon, in which the test becomes hypersensitive due to minimal replicate variance. The extreme precision of the permeability measurement equipment yielded near identical center-point replicates, driving the pure-error variance close to zero. Since this variance serves as the denominator of the LOF calculation, its minute size artificially inflates the F value, causing a false-positive significance result. However, aligned with Quality by Design principles for robust process optimization, the model's high R^2 value (0.9482) confirms that this mathematical anomaly does not compromise its predictive reliability [20, 21].

The results of the significance analysis are shown in Table 2. The model P value < 0.0001 indicates that the relationship among the model variables is extremely significant. The regression equation accurately reflects the relationship between permeability evolution and the thermodynamic factors. Based on the analysis of variance outcomes, the primary hierarchy of influence exerted by the three independent variables on the terminal pyrolysis permeability of the oil shale is: B (final pyrolysis temperature) $>$ A (heating

rate) > C (constant-temperature duration). The extreme statistical significance of variable B directly corroborates the physical mechanistic observation that breaking the thermal activation threshold for kerogen pyrolysis is the absolute dominant driver of extensive fracture generation. This conclusion is consistent with the order of the three response variables in terms of weightless physical properties. The response surface analysis is shown in Figure 5.

Table 2. Regression model variance analysis results

Source	Sum of squares	Degrees of freedom	Mean square	F value	P value	Significance
Model	8.607×10^{-9}	9	9.564×10^{-10}	14.25	<0.0001	Significant
A	1.957×10^{-12}	1	1.957×10^{-12}	1.92	0.2082	
B	4.137×10^{-9}	1	4.137×10^{-9}	61.64	<0.0001	
C	1.290×10^{-10}	1	1.290×10^{-10}	0.029	0.7128	
AB	2.035×10^{-12}	1	2.035×10^{-12}	0.003	0.8667	
AC	1.103×10^{-10}	1	1.103×10^{-10}	1.64	0.2408	
BC	3.626×10^{-11}	1	3.626×10^{-11}	0.54	0.4862	
A2	1.446×10^{-10}	1	1.446×10^{-10}	2.15	0.1856	
B2	4.726×10^{-10}	1	4.726×10^{-10}	7.04	0.0328	
C2	3.410×10^{-11}	1	3.410×10^{-11}	0.51	0.4991	
Lack of fit	4.69	7	6.712×10^{-11}		0.0021	Significant
Pure error	9.077×10^{-9}	16				

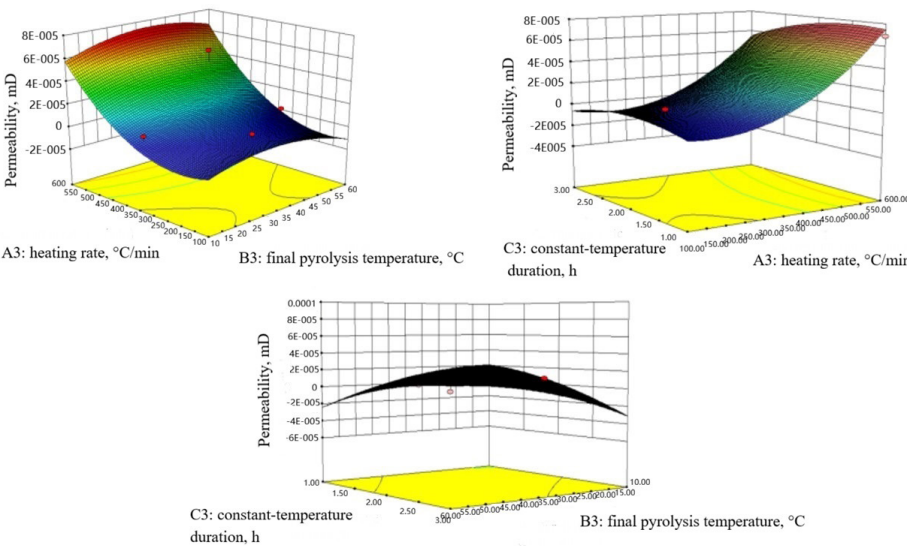


Fig. 5. Response surface analysis of the factors influencing the pyrolysis permeability of oil shale.

3.2. Determination and validation of the optimal heating rate

The optimal pyrolysis environment for the physical parameters of oil shale pyrolysis was obtained by analyzing the experimental results using Design-Expert software. The heating rate should be controlled at 34.55 °C/min, the optimal final temperature of the pyrolysis reaction should be 475 °C, and the duration of maintaining a constant temperature until the final temperature of pyrolysis is 131 min. The predicted permeability value is 7.903×10^{-5} mD.

To verify the validity of the model, based on the actual conditions, the pyrolysis conditions were modified according to the optimized response data with a heating rate of 35 °C/min, a final temperature of 480 °C, and a constant temperature duration of 130 min. Under these conditions, to ensure the validity of the experiment, three pyrolysis experiments were conducted to verify the response values and calculate the average. The experimentally measured post-pyrolysis permeability of the oil shale was 7.075×10^{-5} mD. The extremely high concordance between this physical validation measurement and the theoretical response prediction solidly compensates for the statistical lack-of-fit outcome, providing empirical proof that the regression model is highly reliable for optimizing operational parameters.

4. Conclusions

Fushun oil shale was studied to investigate the effects of different heating rates (10, 20, 30, 40, 50, and 60 °C/min) on the physical properties of Fushun oil shale. First, the pyrolysis characteristics of Fushun oil shale at different heating rates were analyzed. Second, the permeability response was optimized to obtain the optimal heating rate affecting the pyrolysis of oil shale. The following conclusions are drawn:

1. The permeability evolution of Fushun oil shale exhibits a distinct, non-linear dynamic that is heavily dependent on thermal thresholds. Below 300 °C, permeability alterations remain negligible and are primarily pore-structure-dominated, driven by localized mineral expansion and moisture evaporation. Conversely, between 400 °C and 500 °C, the massive decomposition of kerogen induces severe fluid overpressure, violently shifting the matrix into a fracture-dominated regime characterized by exponential permeability enhancement through penetrating fracture networks. Above 500 °C, mineral phase transformations contribute to auxiliary crack propagation.
2. The primary order of the effects of each factor on permeability is final pyrolysis temperature > heating rate > constant-temperature duration
3. Based on the established novel quantitative prediction model, the optimal environmental control parameters were as follows: a heating rate of 34.55 °C/min, an optimal final pyrolysis temperature of 475 °C, and a constant-temperature duration of 131 min. The predicted pyrolysis permeability of the oil shale obtained was 7.903×10^{-5} mD.

Data availability statement

The datasets generated and analyzed during this study are available from the corresponding author upon reasonable request.

Acknowledgments

Yanchao Wang conceptualized the study. Jinjie Huo and Shuangchun Yang validated the methodology, while Jinjie Huo conducted the investigation. Jinjie Huo and Shuangchun Yang provided the resources, and Jinjie Huo and Yi Pan curated the data. Jinjie Huo and Zihao Lu prepared the original draft of the manuscript, which was reviewed and edited by Zihao Lu and Shuangchun Yang. Shuangchun Yang and Yi Pan supervised the study, and Jiabing Zheng revised the manuscript. All authors have read and agreed to the published version of the manuscript. This work was supported by the National Science Foundation of China (NSFC) (grant No. 52574041) and the Liaoning Province Science and Technology Joint Program (2025JH2/101800421). The publication costs of this article were partially covered by the Estonian Academy of Sciences.

Nomenclature

Symbol	Description	Unit
β	Heating rate	$^{\circ}\text{C}/\text{min}$
D	Core diameter	cm
L	Core length	cm
A	Core cross-sectional area	cm^2
Q_0	Actual flow rate	ml/s
Q'_0	Flowmeter reading	ml/min
P_a	Absolute atmospheric pressure	MPa
k_g	Gas permeability	mD
μ	Gas viscosity at the test temperature and atmospheric pressure	$\text{mPa}\cdot\text{s}$
t	Room temperature	$^{\circ}\text{C}$
Y_3	Oil shale pyrolysis permeability	mD
A	Independent variable (heating rate) in response surface optimization	$^{\circ}\text{C}/\text{min}$
B	Independent variable (final pyrolysis temperature) in response surface optimization	$^{\circ}\text{C}$
C	Independent variable (constant-temperature duration) in response surface optimization	h
P_1	Absolute pressure at the core inlet	MPa
P_2	Absolute pressure at the core outlet	MPa

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