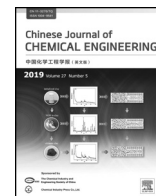




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Article

Numerical simulation and experimental study on dissolving characteristics of layered salt rocks

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ABSTRACT

Underground salt cavern reservoirs are ideal spaces for energy storage. China is rich in salt rock resources with layered lacustrine sedimentary structures. However, the dissolution mechanism of layered salt rocks remains poorly understood, resulting in significant differences between the actual measurements and the designed indices for the layered salt rock water-soluble cavity-making cycle and the cavity shape. In this work, the dissolution rates of 600 groups of layered salt rocks in China under different conditions were determined experimentally. Thus, the established artificial neural network prediction model was used to assess the effects of the contents of NaCl, Na₂SO₄, and CaSO₄ in the salt rocks, concentrations, dissolution angles, and flow rates on their dissolution rates by performing ANOVA and F-test. The results provide a theoretical basis for evaluating the dissolution rate of layered salt rocks under different conditions and for the numerical simulation of the layered salt rock water-soluble cavity-making process.

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1. Introduction

Salt rock deposits with good creep behavior, low permeability (less than 10^{-20} m²), and self-healing property are ideal places for energy (petroleum and natural gas) storage and nuclear waste disposal [1,2]. Storing petroleum and natural gas in salt rock cavities is widely practiced globally [3]. Salt rock is suitable for the water-soluble cavity-making process, in which the dissolution characteristics of salt rock determine the cavity shape.

Different from the thick salt dome formed by marine sediments, China's salt rock presents as a layered structure formed by lake sediments. At present, the mechanism of water-soluble cavity under multi-factor coupling is not clear. Therefore, the countermeasures of the water-soluble cavity-making process for different geological conditions are not formed. Lack of effective control methods in the process and shape of the cavity result in a large difference between the design period of the cavity and the shape of the cavity. The morphology of the cavity is related to the stability of the reservoir, which is directly related to the success of the cavity formation. Therefore, studying the dissolution characteristics of layered salt rock plays an important role in guiding the formation of layered salt rock water-soluble cavities and well mineral salt mining. Whether it is salt rock mining or water-

soluble cavity, salt mine must be solved first. In the salt rock mining process, the yield of salt products increased with the accelerated dissolution of salt rock. The quality of the well mineral salt improved by controlling the concentration of the brine. To accelerate the rate of cavity formation, the dissolution of salt rock should be accelerated, and to ensure the stability of salt cave, the shape of the salt cavity should be controlled.

Considering the boundary layer theory combined with the relevant experimental data on the natural convective dissolution on a smooth vertical salt rock surface, Durie and Jessen [4,5] established a basic equation for the dissolution rate and presented the salt dissolution rate as a key function of brine concentration. In the water-soluble dissolution process, the uneven dissolution on the salt-dissolving surface caused by the heterogeneous rock composition roughens the surface and increases the dissolution area, thus accelerating the dissolution rate. Hans Ulrich Rohr [6] found that in a NaCl-themed multi-component brine system, the dissolution rates of most salts decrease with increasing NaCl concentration. Manvan Alkattan *et al.* [7,8] found that traces of Fe and Zn metals exert an insignificant effect on the salt rock dissolution, whereas Co, Cr, Cd, Pb, F⁻, Br⁻, and I⁻ reduce the dissolution rate of the salt rock. Jesus Guerrero *et al.* [9] proposed that the depression of glauberite inter-bedded with salt rock is often caused by the presence of glauberite. These studies have focused primarily on the mechanics [10,11] and constitutive equations [12–14] of domed salt rock, and on the stability of salt rock reservoirs [15–18]. To the best of our knowledge, no study has explored the dissolution characteristics of layered

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salt rocks. In contrast to domed salt rocks, layered salt rocks are mainly composed of salt rocks, high-salinity mudstone intercalations, glauberite mudstone, mudstone, siltstone, and other similar materials, which are cemented together. As such, layered salt rocks are characterized by complex lithological formations, poor salt rock quality, thinness, and multi-sandwich features [15,19].

The Jintan Salt Mine in Jiangsu Province and the Yunying Salt Mine in Hubei Province were selected as study areas to investigate the dissolution rates under different conditions and the dissolution properties of layered salt rocks. The dissolution rates of 600 groups of salt rocks under different conditions were determined experimentally. In addition, an artificial neural network model was established to simulate the effects of the contents of NaCl, Na₂SO₄, and CaSO₄ in salt rocks, concentrations, dissolution angles, and flow rates on their dissolution rates. ANOVA and F-test were conducted to verify the results. The findings of this study provide a theoretical basis for evaluating the dissolution rate of layered salt rocks under different conditions and for numerically simulating the layered salt rock water-soluble cavity-making process.

2. Salt Rock Dissolution Mechanisms

Salt rock dissolution is a non-selective process. Salt minerals are inherently easily soluble in water. When water comes in contact with salt minerals, the ions that make up the crystal lattice are attracted by the oppositely charged end of the water molecules. When the gravitational pull of the water molecules toward the ions is sufficient to overcome the gravitational attraction among the ions, then the crystal lattice will be damaged by the salt minerals. Thus, the ions enter the water, and the salt minerals are dissolved.

In view of the compactness of salt minerals, dissolution mainly occurs on the mineral surface; that is, minerals are gradually dissolved from the outside to the inside. At the initial dissolution stage, the solution has an extremely low salinity, and the minerals are dissolved quickly. Although the solution concentration around the mineral surface gradually increases over time, the salt-dissolving and salt-receiving capacities of the solution decrease, resulting in a decreased dissolution rate. However, the solution concentration away from the mineral surface remains low. That is, a certain concentration difference exists between the solutions around and away the mineral surface. According to the solute diffusion principle, such concentration difference causes salt minerals to diffuse from highly concentrated brine areas to poorly concentrated areas. Thus, the solution concentration around the mineral surface increases its capacity to continuously dissolve, and the diffusion continues until the entire solution is saturated. This phenomenon is the solute diffusion during the salt mineral dissolution.

As shown in Fig. 1, the dissolution process includes the following scenarios. ① In the dissolved boundary area, salt rock molecules on the boundary diffuse to the diffusion area under the influence of the diffusion gradient due to the concentration difference, resulting in a

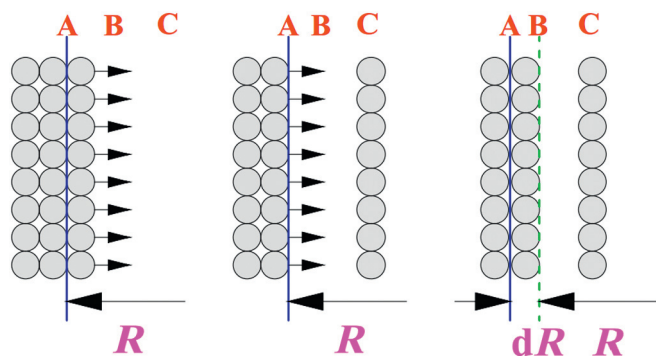


Fig. 1. Salt rock boundary dissolved diagram. (A - the salt rock surface, B - the bottom of the boundary, C - the diffusion area)

decreased concentration of the bottom layer that is even below the saturated concentration. ② Salt molecules on the solid salt rock surface are dissolved and proceed to the bottom of the boundary, thereby maintaining the concentration balance and ensuring that the concentration of the underlying solution achieves a new dynamic equilibrium. ③ For dissolving cavities, salt rock dissolution enlarges the cavity radius dR and the total radius $R + dR$, and the solution reaches a new dynamic equilibrium on the underlying cavity surface and the diffusion area.

3. Experiment

3.1. Materials

All layered salt rocks used in the experiment were provided by Langfang Branch of the Exploration and Development Institute of Petro China and were mainly sourced from Jintan, Jiangsu, and Yunying, Hubei. To ensure that the salt rock dissolution area remained unchanged, the core obtained during the exploration was cut, sealed with wax, and processed into pieces for dissolution test, as shown in Fig. 2.

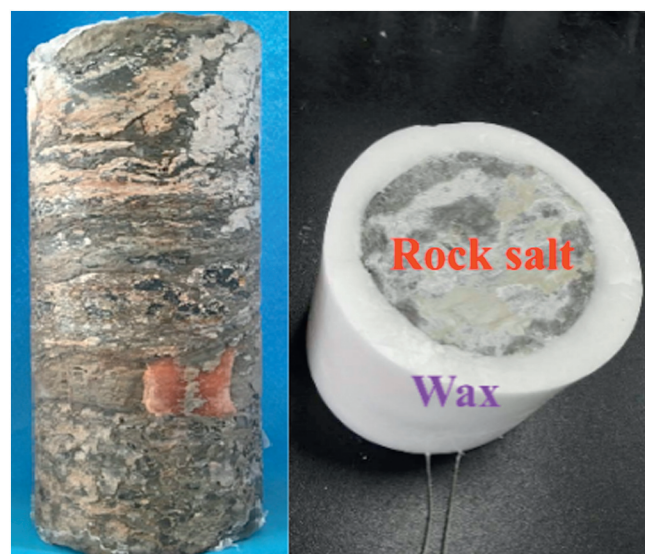


Fig. 2. The layered Salt Rock and the salt rock test piece.

3.2. Experimental methods

Salt rock dissolution rate is important parameters for characterizing salt dissolution characteristics. The salt of dissolved salt minerals per unit time and unit area is called rock salt dissolution rate.

The experimental installation is depicted in Fig. 3. In the experiment, after the temperature of 10 L NaCl solution at a certain concentration was adjusted to 55 °C, the salt rock test pieces were dissolved in different ways and collected. Then, the dissolution rate was calculated by using Eq. (1) after the changes in the solution concentration were determined. In the experiment, the dissolution angle of the salt rock sample was controlled by adjusting the length of the suspension rope and was measured by a protractor. The flow rate of the solution flowing through the salt rock test piece was adjusted by a valve and was calculated by the data measured by the flow meter. The salt rock test piece was cut and wax sealed to maintain a constant end face during the dissolution process. Therefore, the salt rock dissolution area was determined by measuring the size of the dissolved end face of the salt rock test piece.

$$\nu = \frac{C_2 V_2 - C_1 V_1}{S \cdot t} \quad (1)$$

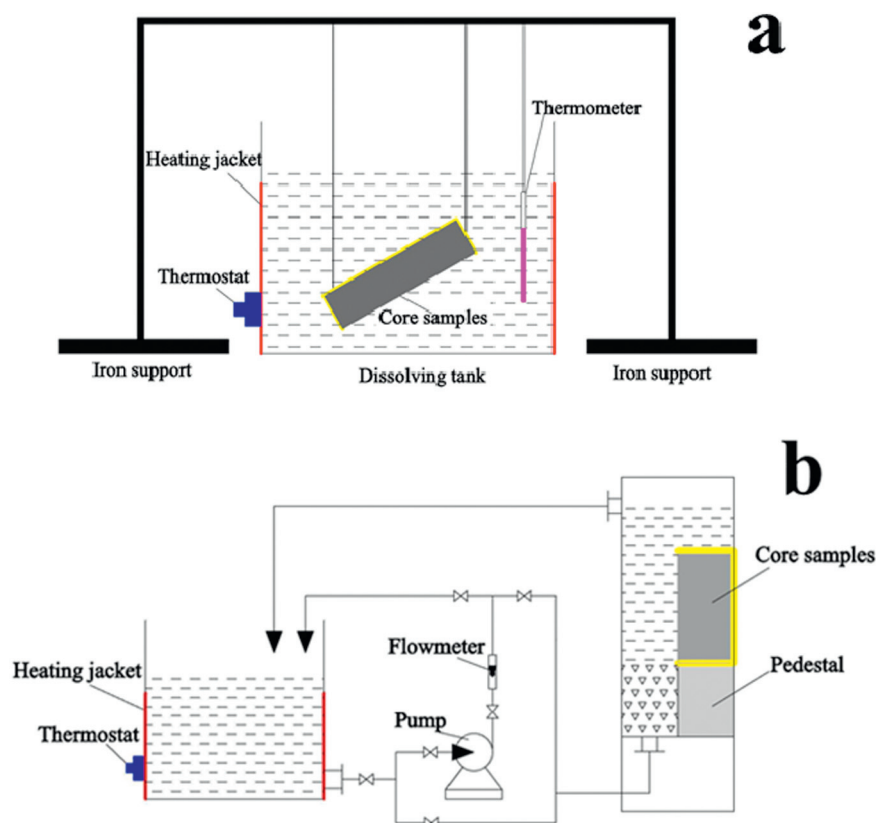


Fig. 3. Schematic diagram of experimental equipment for salt rock dissolution. (a - static dissolved, b - dynamic dissolved)

where ν denotes the salt rock dissolution rate; C_1 and C_2 denote the solution concentrations before and after dissolution; V_1 and V_2 denote the volumes of the solution before and after dissolution (the differences in volume before, during, and after the dissolution were omitted, $V_1 \approx V_2$); S denotes the dissolved area of the salt rock, and t denotes the time spent on dissolving the salt rock.

Chloride, calcium, and sulfate ions were measured by standard silver nitrate titration, standard EDTA titration, and barium sulfate gravimetry, respectively. The BP artificial neural network model on MATLAB software was utilized mainly for training the data processing of the simulation.

The variation ranges of all factors are listed in Table 1.

Table 1
The variation ranges of dissolution factors

Parameters	Minimum	Maximum
NaCl in rock salt, $X_1/\%$	1.86	99.02
CaSO ₄ in rock salt, $X_2/\%$	0.00	15.26
Na ₂ SO ₄ in rock salt, $X_3/\%$	0.00	16.10
Insoluble substance in rock salt, $X_4/\%$	0.28	85.04
NaCl in the solution, $C/\text{g} \cdot \text{L}^{-1}$	0	313
Dissolution angle, $\alpha/(\text{°})$	0	180
Flow rate, $\nu/\text{m} \cdot \text{s}^{-1}$	0	0.02

4. Results and Discussion

4.1. Experimental data

Because the composition of the layered salt rock is quite different, the corresponding single factor experiment cannot be carried out in the experiment. Moreover, there are many factors influencing the

experiment, and it has nonlinear characteristics, which make it difficult to simulate with traditional methods. Artificial neural networks are powerful tools for describing and dealing with multi-factor nonlinear complex problems. An artificial neural network model was established by measuring a large amount of dissolution rate data. The effects of various factors on the dissolution rate of salt rock were studied according to the established artificial neural network model. In the experiment, about 600 sets of dissolution rate data were measured under different conditions.

4.2. Data processing

Layered salt rock is difficult to simulate through traditional methods because of the significant differences in the compositions, influencing factors, and nonlinear characteristics. Artificial neural network is a powerful tool for describing and handling complex multi-factorial nonlinearities [20,21]. In the experiment, on MATLAB software was utilized to establish a BP artificial neural network that was a multi-layer forward neural network for one-way propagation and an algorithm for error back-propagation. The BP algorithm is the most widely used and most representative neural network learning algorithm in recent years [22,23]. In the data processing, 600 datasets were divided into three groups: 70% for training; 15% for verification; and 15% for testing. The neurons of BP learning algorithm have been divided into three parts: an input layer, a hidden layer, and an output layer network. The ANN architecture has a configuration of 5–9–1 neurons. In the establishment of BP artificial neural network in MATLAB software, the hidden layer activation function used Logarithmic sigmoid transfer function (the string of the function is *logsig*), and the output layer activation function used linear transfer function (the string of the function is *purelin*). The training function used the momentum reduction and *traingdx*, a dynamic adaptive learning rate gradient descent BP algorithm training

function, and the learning function selected *learnngdm*, a BP learning rule with the momentum item.

The established artificial neural network model presented good results, as evidenced by the correlation coefficient R^2 of over 0.95 for the training, verification, and testing data, and the root mean square error of below 5. According to the error curve, it can be determined that there is no overfitting in the training process.

4.3. Changing regularities of the salt rock dissolution rate

4.3.1. Effects of the dissolution angle on the dissolution rate

The static dissolution rate was experimentally simulated with a concentration of $0 \text{ g} \cdot \text{L}^{-1}$ at a temperature of 55°C and a salt rock dissolution angle of 0° – 180° . As shown in Fig. 4, the dissolution rate generally agreed with the rule that the dissolution rate increased with the dissolution angle. This effect was mainly related to the gravitational pull of the soluble salt particles on the dissolved salt rock surface and the attraction of other particles in the salt rock. Salt rock dissolution is a process in which the soluble salt particles in the ore come into contact with the water and leaves from the ore interface under its induction. The vertical downward gravitational pull of particles and the attraction between water molecules and the salt rock in different directions relatively to the dissolved surface vary with the spatial position of the dissolved surface of salt rock. Accordingly, the speed at which particles leave from the lattice and its surrounding area, that is, the dissolution rate, is also different. Therefore, a cushion protection is required on the top of the cavity during the actual salt rock water-soluble mining to prevent the top of the cavity from being in direct contact with the brine.

4.3.2. Effects of the flow rate on the dissolution rate

The dissolution rate at 55°C was experimentally simulated in fresh water, $100 \text{ g} \cdot \text{L}^{-1}$ NaCl solution, and $200 \text{ g} \cdot \text{L}^{-1}$ NaCl solution at different flow rates (0 – $0.02 \text{ m} \cdot \text{s}^{-1}$). As shown in Fig. 5, the dissolution rates of the corresponding test pieces increased with increasing flow rate regardless of the dissolution concentration. Therefore, an increase in the flow rate may accelerate the dissolution of the salt rock primarily because of the multi-factorial interaction process of the salt rock in the solution, which includes physical dissolution, chemical leaching, convective diffusion, and heat-mass transfer. The flow of the solution

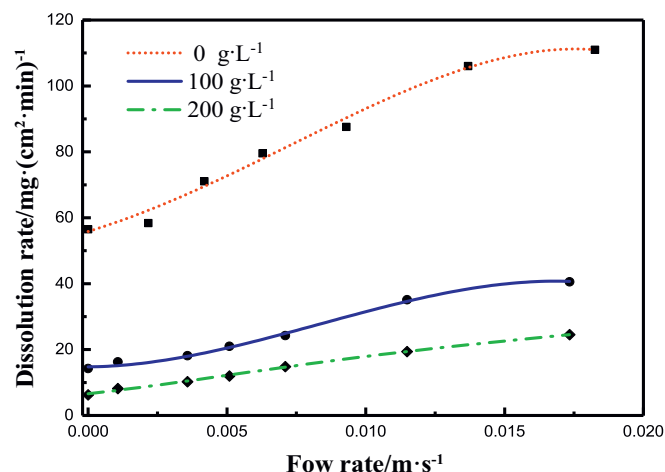


Fig. 5. The dissolution rate in function of the flow rate.

exerted a hydraulic scouring effect on the salt rock. The solution had a uniform concentration. The flow of the solution accelerated the convection and diffusion of salt ions in the solution, and increased the dissolution rate. The dissolution rate of the minerals varied with the flow rate.

4.3.3. Effects of the salt rock composition on the dissolution rate

For the salt rock composition, the NaCl content varied within the range of 10%–90% to ensure that the salt rock had the same content of Na_2SO_4 as that of CaSO_4 (i.e., 5%). The static dissolution rate of the salt rock at different concentrations was experimentally simulated with a dissolution angle of 180° and a temperature of 55°C . As shown in Fig. 6, the dissolution rate initially increased with increasing NaCl content in the salt rock and then gradually decreased with increasingly higher NaCl concentrations in the solution. The dissolution rate in fresh water with 90% NaCl content was approximately 14.8 times of that in fresh water with 10% NaCl content. The reason is that the main component of the soluble salt in the salt rock was NaCl, of which the dissolution rate was directly related to the total dissolution rate of the salt rock.

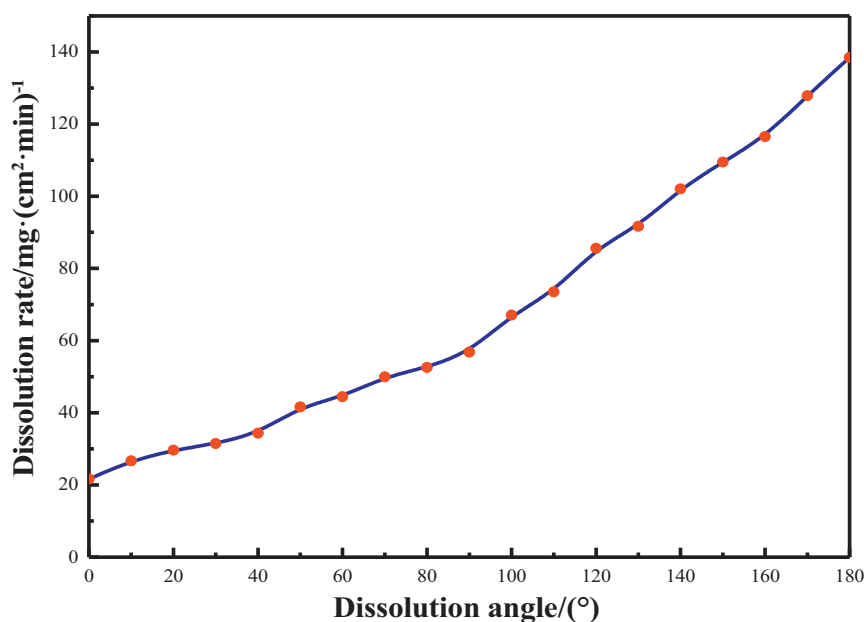


Fig. 4. The dissolution rate in function of the dissolution angle.

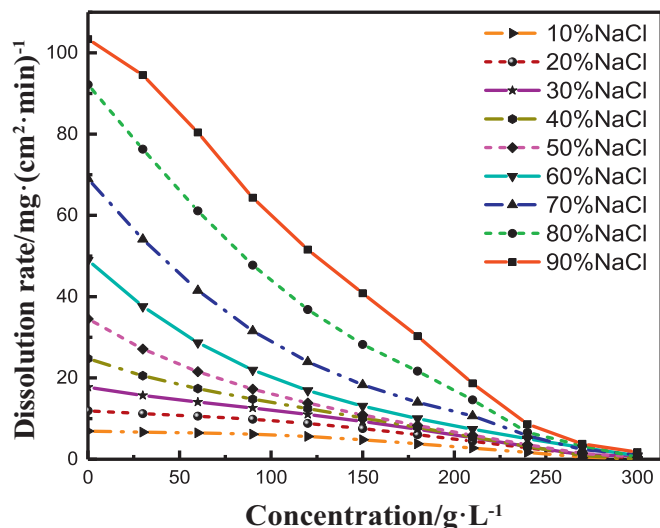


Fig. 6. Effects of the contents of NaCl in salt rocks on the dissolution rate.

Salt rock is composed of Na_2SO_4 with content varying in the range of 0%–15%. To ensure that the salt rock had the same content of NaCl and CaSO_4 , NaCl accounted for 60%, and CaSO_4 accounted for 5%. The static dissolution rates of such a salt rock composition at different concentrations were experimentally simulated at a dissolution angle of 180° and a temperature of 55°C . As shown in Fig. 7, the dissolution rate exhibited a down-and-up trend with increasingly higher contents of sodium sulfate in the salt rock. When the sodium sulfate content was 6%, the total dissolution rate reached the valley. The dissolution rate in fresh water with 6% sodium sulfate in fresh water was at least 12.3% lower than that of salt rock without sodium sulfate, and the dissolution rate in salt rock with 15% sodium sulfate content was higher than that without sodium sulfate. Given that sodium sulfate is another important soluble salt apart from NaCl in salt rock, when the salt rock contains a very small amount of sodium sulfate, sodium sulfate may appear in the solution and change the original solution system. As the ternary phase diagram of $\text{Na}^+//\text{Cl}^-$, SO_4^{2-} – H_2O (Fig. 8), if any amount of sodium sulfate is present in the solution, the NaCl solubility drops from 26.8% to 24.2%, thus reducing the dissolution impetus of the main soluble salt – NaCl – and decreasing the dissolution rate of NaCl. However,

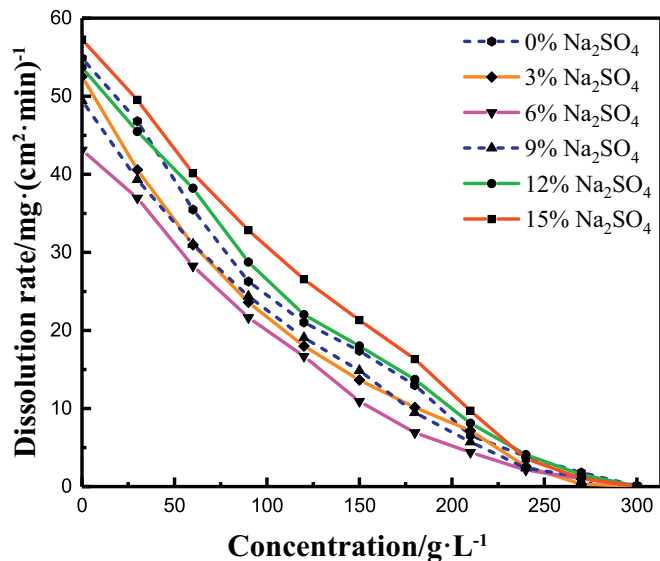


Fig. 7. Effects of the contents of Na_2SO_4 in salt rocks on the dissolution rate.

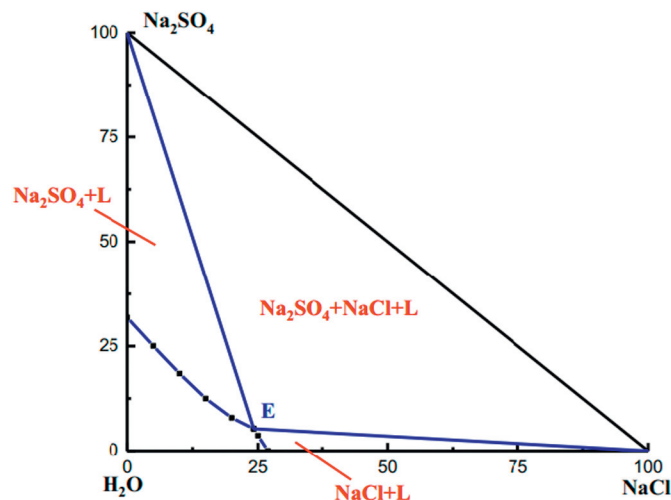


Fig. 8. Phase diagram of $\text{Na}^+//\text{Cl}^- \cdot \text{SO}_4^{2-}$ – H_2O system at 50°C .

given that the sodium sulfate content was relatively low at that moment, the dissolution rate was insufficient compensate for the reduced dissolution rate of NaCl. Thus, the total dissolution rate was decreased. When the sodium sulfate content in salt rock reached certain value, the dissolution rate of sodium sulfate became sufficiently large to compensate for the reduced dissolution rate of NaCl. Consequently, the total dissolution rate increased.

For the salt rock composition with CaSO_4 content varying in the range of 0%–15%, NaCl accounted for 60%, and CaSO_4 accounted for 5%. The static dissolution rates of the salt rock at different concentrations were experimentally simulated at a dissolution angle of 180° and a temperature of 55°C . As shown in Fig. 9, the dissolution rate remained unchanged even though calcium sulfate content kept changing. That is, even though other soluble salts, such as NaCl and sodium sulfate, had the same content in the salt rock, the calcium sulfate content only slightly affected the total dissolution rate. The contents of these salts gradually decreased with the increasingly higher concentrations of NaCl in the solution, primarily because the solubility of the calcium sulfate in water was only $2\text{ g}\cdot\text{L}^{-1}$. The low dissolution impetus in the solution caused the extremely low dissolution rate of the calcium sulfate. Compared with the other two kinds of soluble salts (NaCl and

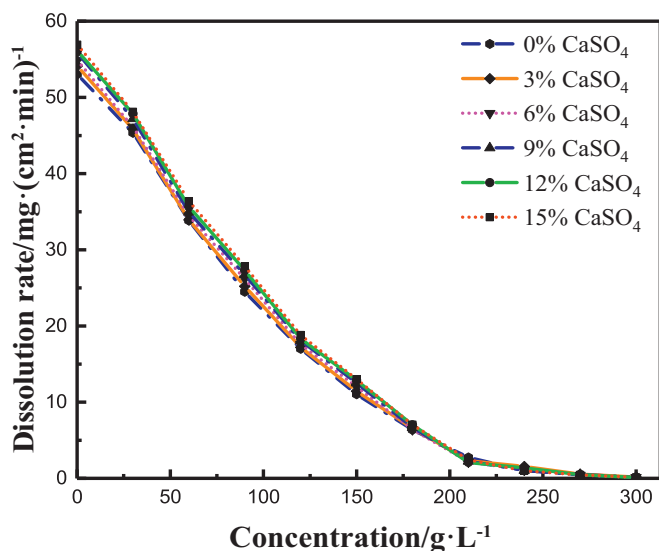


Fig. 9. Effects of the contents of CaSO_4 in salt rocks on the dissolution rate.

Na₂SO₄) displayed negligible dissolution rates. When the salt rock had the same content of NaCl and Na₂SO₄, because the increase in the dissolution rate with the calcium sulfate content was disregarded, the total dissolution rate hardly changed with increasingly higher calcium sulfate contents.

4.3.4. Effects of the solution concentration on the dissolution rate

As shown in Figs. 6, 7, and 9, the dissolution rate decreased with the NaCl concentration regardless of the salt rock composition. This phenomenon is related to the dissolution impetus of the salt rock, and the driving force for the salt rock dissolution process was the difference between the salt solubility and the solution concentration. From a chemical kinetic point of view, such difference is the chemical potential at which the salt rock dissolves. Comparison revealed that when the NaCl concentration reached about 225 g·L⁻¹, the dissolution rate became smaller (close to zero) regardless of any difference in the salt rock compositions, because the salt rock dissolution was limited by the dissolution impetus.

4.4. Multivariate analysis of variance

The obtained *p*-value and *F*-ratio can be used to judge if the effect of experimental factor on response is significant. The effect of experimental factor on response is more significant with the larger *F*-ratio or smaller *p*-value. On the contrary, the response is not affected by factors significantly with the smaller *F*-ratio or larger *p*-value. The degree of response affected by factors was defined by Montgomery to assess the test results of *p*-value, which were listed as follows.

- (1) *p* value > 0.10, insignificant.
- (2) 0.05 < *p* value < 0.10, significant slightly.
- (3) 0.01 < *p* value < 0.05, significant.
- (4) 0.001 < *p* value < 0.01, very significant.
- (5) *p* value < 0.001, extremely significant.

The primary and secondary relations among the various factors affecting the dissolution rate were identified by ANOVA. Table 2 presents the factor levels based on the investigated six influencing factors, namely, the contents of NaCl, Na₂SO₄, and CaSO₄ in salt rock, the solution concentration, the dissolution angle, and the flow rate of the solution. The orthogonal table L18 (3⁷) was selected, and the corresponding orthogonal test results were obtained by using the established artificial neural network prediction model for layered salt rock. Table 3 provides the ANOVA table of each factor.

Table 2
The factors and levels table

Level	NaCl in rock salt /%	Na ₂ SO ₄ in rock salt /%	CaSO ₄ in rock salt /%	NaCl in the solution / g·L ⁻¹	Dissolution angle / (°)	Flow rate / m·s ⁻¹
1	10	5	5	100	0	0.005
2	40	10	10	200	90	0.01
3	70	15	15	300	180	0.015

Table 3
The analysis of variance table

Source	Sum of square (SS)	Degree of freedom (df)	Mean square (MS)	<i>F</i> -ratio	<i>p</i> value
NaCl in rock salt	1607.84	2	803.92	15.50	0.00719
Na ₂ SO ₄ in rock salt	981.90	2	490.95	9.47	0.01995
CaSO ₄ in rock salt	372.62	2	186.31	3.59	0.10786
NaCl in the solution	1994.04	2	997.02	19.23	0.00449
Dissolution angle	1209.00	2	604.50	11.66	0.01311
Flow rate	814.31	2	407.15	7.85	0.02867
Error	259.30	5	51.86		

The findings suggested that the NaCl content in the salt rock and the solution concentration very significantly affected the dissolution rate. The Na₂SO₄ content in the salt rock, the dissolution angle, and the flow rate of the solution exerted significant effects on the dissolution rate. The CaSO₄ content in the salt rock insignificantly influenced the dissolution rate. The factor that most dominantly influenced the dissolution rate was the solution concentration, followed by the NaCl content in the salt rock, the dissolution angle, the Na₂SO₄ content in the salt rock, the flow rate of the solution, and the CaSO₄ content.

5. Conclusions

In this work, dissolving mechanism was determined by investigating the dissolution rates of layered salt rock in China, which can guide the water-soluble mining and cavity-making process of layered salt rock. The effects of the contents of NaCl, Na₂SO₄, and CaSO₄ in the salt rock, the solution concentration, the dissolution angle and the flow rate of the solution on the dissolution rate of layered salt rock were identified. The results revealed that a larger dissolution angle corresponded to a higher dissolution rate, and that an increase in the flow rate of the solution may accelerate the salt rock dissolution. Moreover, the NaCl concentration in the solution may inhibit the salt rock dissolution. The effects of the dissolution angle of salt rock can guide the addition and removal of the protective cushion in the water-soluble cavity-making. ANOVA and *F*-test indicated that the dissolution rate was affected by the following factors in descending order: the solution concentration, the NaCl content in the salt rock, the dissolution angle, the Na₂SO₄ content, the flow rate of the solution, and the CaSO₄ content. This study proposes to predict the cavity-making cycle prior to the water-soluble cavity-making process by evaluating the dissolution rate based on the proven salt rock composition; to establish an important reference for the site selection of salt cavern reservoirs; and to adjust the water inlet flow, the distance between the water inlet and the brine outlet, and the position of the water inlet at the different stages of cavity-making to regulate the concentration and speed distribution of the cavity and the cavity shape in the water-soluble cavity-making process.

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