Effect of Freeze-thaw Cycles on Microstructure and Mechanical Properties of Limestone

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Abstract: In studying the effect of freeze-thaw cycles on the microstructure and mechanical properties of limestone, scanning electron microscope, low-field nuclear magnetic resonance, and uniaxial compression tests are carried out on the limestone samples dried and subject to 5, 10, 15, and 20 freeze-thaw cycles. Through analysis and discussion, it was found that the number of freeze-thaw cycles has a significant effect on the microstructure of the limestone samples. Compared to the dry samples, the number of microcracks of the limestone samples subjected to freeze-thaw cycles is increased. Moreover, the crack width is positively correlated to the number of freeze-thaw cycles. With an increase in freeze-thaw cycles, the signal intensity of the T2 spectrum is considerably increased, and the connectivity of the micropores and fissures is increased. In addition, the peak compressive strength and elastic modulus showed a significant negative correlation with the number of freeze-thaw cycles. Again, compared to the dry samples, the peak compressive strength of the samples subject to 5, 10, 15, and 20 freeze-thaw cycles decreased by 14.7%, 22.0%, 28.7%, and 37.5%, respectively. The elastic modulus also decreased by 8.0%, 11.4%, 11.7%, and 21.5%, respectively. Therefore, these findings provide basic parameters for the stability evaluation of rock slopes in Northwest Guizhou.

Keywords: freeze-thaw cycle; limestone; mechanical property; uniaxial compression; microstructure

Introduction

In recent years, Guizhou Province has invested heavily in infrastructure construction, a large number of rock mass projects have been implemented, and many rock slopes have been formed. The stability of these slopes is critical to the implementation of those projects. The Bijie region is 457–2900 meters above sea level, most of which is high altitude. In the high-altitude areas, freezing weather often occurs in winter. Since the chilly weather is often accompanied by cloudy and continuous rainfall, it significantly impacts the stability of the slopes [1-3]. Therefore, it is of particular theoretical and practical significance to study macro and microstructures and mechanical properties of typical rocks in this area under the action of freeze-thaw cycles.

For a long time, many researchers have devoted themselves to studying the influence of environmental temperature changes on rock mechanics and have achieved worthwhile results. Mousavi et al. carried out the experiments of 0, 7, 15, 40, and 75 freeze-thaw cycles on schist and analyzed the impact of freeze-thaw cycles on its microstructure and triaxial compressive strength [4]. Moreover, Sun Yong et al. analyzed the permeability of coal
subjected to different freeze-thaw cycles using low-field Nuclear Magnetic Resonance (NMR) equipment [5]. One of the essential properties of mudstone is that it swells quickly after absorbing water; therefore, Zeng Zhixiong et al. studied the swelling stress and strain characteristics of mudstone subjected to dry-wet cycles and freeze-thaw cycles [6]. Zhou Keiping and Li Jierin et al. examined the pore structure characteristics of sandstone, granite, and other rocks under the action of freeze-thaw cycles using low-field NMR equipment [7-10]. Zhao Tao et al. [11] and Xi Jiamei et al. [12] used sandstone and medium-grained sandstone from Xinzhuang Coal Mine in Gansu Province as the research sample. After one freeze-thaw cycle, they analyzed the water absorption, microstructure, uniaxial compressive strength, and elastic modulus of the two kinds of sandstones. Liu Haikang et al. conducted freeze-thaw deterioration tests on sandstones with different initial water contents and analyzed the changes in water absorption, longitudinal wave velocity, uniaxial compressive strength, and other parameters of sandstone in the comparison to the dry group, saturated group, and saturation comparison group [13]. Lu Yani et al. [14] considering fractured rock mass in a cold region as the research focus, made fractured rock samples with different geometric characteristics by using cement-mortar rock materials, carried out freeze-thaw cycle tests and uniaxial compression tests on prefabricated fractured rock samples of distinct features, and examined the effect of cycle count on its permeability. Liu Shaohe et al. [15] performed impact tests on red sandstone specimens subjected to different freeze-thaw cycles by using separated Hopkinson pressure bars with a diameter of 100 mm, and obtained the stress-strain curves of the red sandstone after each cycle, and investigated the changes in peak stress, peak strain, and elastic modulus. Zhang Huimei et al. [16] conducted mechanical property tests on saturated red sandstone subjected to 0, 5, 10, 20, and 40 freeze-thaw cycles at four different confining pressures and analyzed the influence of freeze-thaw cycles and the confining pressure on the physical and mechanical properties of red sandstone. Ni Xiaohui et al. [17] and Wang Lehua et al. [18] used the surrounding rock sandstone samples and bedding sandstone from tunnels in cold areas as research objects, and conducted confining pressure unloading experiments on them after different freeze-thaw cycles, and discussed the strain energy transformation characteristics and mechanical properties.

In summary, the previous studies on the influence of environmental temperature on rocks’ physical and mechanical properties are mainly focused on the Qinghai-Tibet Plateau and Sichuan-Tibet Railway-related engineering projects. There is relatively little research on the slopes and foundations, tunnels, mines, and hydropower projects conducted for the Yunnan-Guizhou Plateau, especially the Bijie region. During the seasonal and day-night alternations, the engineering of rock mass in the Yunnan-Guizhou Plateau faces irreversible damage such as low-temperature frostbite, which will have an uncertain impact on the safety and stability of slopes, foundation pits, tunnels, mines, and hydropower projects. Therefore, the study of the microstructure and mechanical properties of limestone subjected to freeze-thaw cycles is of important academic value and practical significance for evaluating the safety and stability of rock engineering and the design, support, and construction of rock engineering.

1 Sample Collection and Processing and Experimental Scheme

1.1 Sample Collection and Processing

Limestone samples are collected in the Xinhua District of Bijie City and transported to the university laboratory. According to the required specification [19], standard samples of Φ50 mm×100 mm are made by coring, cutting, and grinding the limestone with a coring machine, a cutting machine, and a grindstone machine. A total of 15 standard samples are divided into five groups, labeled; Group A (dry sample), Group B (5 freeze-thaw cycles), Group C (10 freeze-thaw cycles), Group D (15 freeze-thaw cycles), and Group E (20 freeze-thaw cycles). Then the samples used
for scanning electron microscope (SEM) tests are processed into cuboids of length (1 cm) × width (1 cm) × thickness (0.5 cm). Finally, the samples used for low-field NMR tests are processed into cylinders of Φ50 mm×100 mm, as shown in Fig. 1.

1.2 Experimental Scheme

To examine the influence of freeze-thaw cycles on limestone’s microscopic structure and mechanical properties, the following freeze-thaw scheme is formulated with respect to the temperature difference between day and night (about 10°C in the daytime and −2°C at night) and the weather conditions in the Bijie region. First, the limestone samples of Group B, C, D, and E are immersed in distilled water for natural saturation for seven days. Next, the four groups of samples are frozen in a controlled refrigerator for 12 hours and then thawed in water for 12 hours. Then, the limestone samples of Group B, C, D, and E are subjected to 5, 10, 15, and 20 freeze-thaw cycles, respectively. After that, the samples are naturally dried for 10 days. Scanning electron microscope tests, low-field NMR tests, and uniaxial compression tests are performed using the FEI-SEM equipment and NMR equipment RMT-150C mechanical test system from Henan Polytechnic University. Finally, the mechanical experiment adopts the displacement-controlled loading method, at a rate of 0.002 mm/s.

2 Effect of Freeze-thaw Cycles on Physical Parameters of Limestone

The density and wave velocity of limestone samples were measured before and after freeze-thaw cycles with ultrasonic testers, balances, calipers, and other instruments to investigate the effect of the number of freeze-thaw cycles on the density and wave velocity of limestone in the Bijie region. The basic parameters of limestone subjected to various freeze-thaw cycles are shown in Table 1. Before and after freeze-thaw cycles, the wave velocities of the samples in Group B (5 freeze-thaw cycles) are 5636 m/s and 4605 m/s, respectively. After five freeze-thaw cycles, the wave velocity is decreased by 18.3%. The wave velocities of Group C (10 freeze-thaw cycles) before and after freeze-thaw cycles are 5554 m/s and 4251 m/s, respectively. After 10 freeze-thaw cycles, the wave velocity is decreased by 23.5%. The wave velocities of Group D (15 freeze-thaw cycles) before and after freeze-thaw cycles are 5448 m/s and 3807 m/s, respectively. After 15 freeze-thaw cycles, the wave velocity is decreased by 30.1%. The wave velocities of Group E (20 freeze-thaw cycles) before and after freeze-thaw cycles are 5357 m/s and 2772 m/s, respectively. After 20 freeze-thaw cycles, the wave velocity decreased by 48.3%. Therefore, it is evident from the above analysis and Table 1 that the number of freeze-thaw cycles has no significant effect on the density of limestone but has a considerable effect on the longitudinal wave velocity of limestone. The reason for this observation being that the water in the limestone freezes into ice, expands in volume, and forms new cracks. The existence of these
cracks hinders the propagation of ultrasonic waves. Therefore, the wave velocity decreases obviously after the freeze-thaw cycle. However, due to the excellent cementation and high strength of limestone, the mass and volume of the limestone sample remain unchanged during the freeze-thaw process. Thus, rendering its density unchanged.

### Tab. 1 Basic parameters of limestone subjected to different freeze-thaw cycles

| No. | Number of freeze-thaw cycles | Height/mm | Diameter/mm | Wave velocity before freezing and thawing/(m.s\(^{-1}\)) | Wave velocity after freezing and thawing/(m.s\(^{-1}\)) | Density before freezing and thawing/(kg.m\(^{-3}\)) | Density after freezing and thawing/(kg.m\(^{-3}\)) |
|-----|-----------------------------|-----------|-------------|--------------------------------------------------------|--------------------------------------------------------|------------------------------------------------|------------------------------------------------|
| A-1 | 0                           | 99.60     | 49.56       | 5242                                                   | --                                                     | 2718                                           |                                                   |
| A-2 | 0                           | 99.82     | 49.50       | 5485                                                   | --                                                     | 2721                                           |                                                   |
| A-3 | 0                           | 100.00    | 49.60       | --                                                     | --                                                     | 2717                                           |                                                   |
| B-1 | 5                           | 99.92     | 49.62       | 5677                                                   | 4850                                                   | 2709                                           | 2709                                           |
| B-2 | 5                           | 99.62     | 49.54       | 5565                                                   | 4447                                                   | 2716                                           | 2715                                           |
| B-3 | 5                           | 100.30    | 49.56       | 5667                                                   | 4518                                                   | 2706                                           | 2706                                           |
| C-1 | 10                          | 99.92     | 49.60       | 5511                                                   | 4307                                                   | 2687                                           | 2689                                           |
| C-2 | 10                          | 100.00    | 49.50       | 5495                                                   | 4167                                                   | 2714                                           | 2716                                           |
| C-3 | 10                          | 100.10    | 49.62       | 5655                                                   | 4278                                                   | 2714                                           | 2711                                           |
| D-1 | 15                          | 99.98     | 49.62       | 5524                                                   | 3890                                                   | 2704                                           | 2703                                           |
| D-2 | 15                          | 100.10    | 49.62       | 5500                                                   | 3895                                                   | 2698                                           | 2700                                           |
| D-3 | 15                          | 100.00    | 49.60       | 5319                                                   | 3636                                                   | 2703                                           | 2704                                           |
| E-1 | 20                          | 99.62     | 49.62       | 5414                                                   | 2790                                                   | 2720                                           | 2717                                           |
| E-2 | 20                          | 99.82     | 49.54       | 5338                                                   | 3007                                                   | 2713                                           | 2704                                           |
| E-3 | 20                          | 100.00    | 49.62       | 5319                                                   | 2519                                                   | 2702                                           | 2702                                           |

### 3 Effect of Freeze-thaw Cycles on Microstructure Characteristics of Limestone

#### 3.1 Analysis of Energy Spectrum Characteristics of Limestone

To analyze the composition characteristics of limestone, 5 collection points of the limestone sample are selected for energy spectrum analysis using the energy spectrometer during the SEM test, as shown in Fig. 2. Moreover, Fig. 3 shows the energy spectrum characteristics of the 5 collection points of the sample in Group A. As can be seen from Fig. 3, the main components of the limestone sample include calcium carbonate, iron oxide (Fe\(_x\)O\(_y\)), feldspar, quartz, and the likes.

![Fig. 2 Five collection points on limestone sample for energy spectrum analysis](image-url)
3.2 Analysis of Influence of Freeze-thaw Cycles on Microstructure Characteristics of Limestone

Fig. 4 shows the microstructure photos of the limestone samples subjected to 5, 10, 15, and 20 drying and freeze-thaw cycles. It can be seen from the figure that there are no cracks in the dried sample. With the gradual increase of the number of freeze-thaw cycles, the microscopic particles of the limestone sample begin to crack under the action of repeated freezing and water expansion, and the number and width of the cracks increase accordingly. To appreciate the influence of freeze-thaw cycles on the number and width of microscopic cracks in limestone, all the samples are first collected with FEI-SEM. Then the number of microscopic cracks are analyzed through the scanning microscope image analysis software; SmileView, as shown in Fig. 5. Fig. 6 shows the statistical analysis of the width and number of microscopic cracks in the limestone samples subjected to 5, 10, 15, and 20 drying and freeze-thaw cycles. This figure indicates that the number of cracks in the dry sample is minimal, only 2, and the average crack width is 0.435 um. After 5 freeze-thaw cycles, the number of cracks in the limestone sample increases significantly to 36, and the crack width range is also markedly expanded from 0 to 0.8 um. After 10 freeze-thaw cycles, the number of cracks in the limestone sample continued to increase to 63. Although the range of crack width does not increase significantly, cracks with larger width appear. After 15 freeze-thaw cycles, the number of cracks in the limestone sample increases, but only by 5. However, the range of crack width is expanded obviously from 0 to 1.2 um. After 20 freeze-thaw cycles, the number of cracks in the limestone sample is reduced to 38, but the crack width is increased significantly, reaching a maximum of 2.4 um. Indicating that after 20 freeze-thaw cycles, some cracks in the limestone converge and connect, forming larger cracks.
Fig. 4 Microstructure of limestone samples subjected to different freeze-thaw cycles.

Fig. 5 Measurement and analysis of micro cracks in limestone sample after 15 freeze-thaw cycles.
3.3 Analysis of Influence of Freeze-thaw Cycles on Pore Structure Characteristics of Limestone

To understand the impact of freeze-thaw cycles on the pore structure characteristics of the limestone samples, T2 spectroscopy tests are performed on the limestone samples subjected to 5, 10, 15, and 20 drying and freeze-thaw cycles with low-field NMR equipment. The low-field NMR T2 spectrum can reflect the porosity and pore size of the limestone sample. The value of T2 is positively correlated with the pore size, whereas the area of the T2 spectrum is positively correlated to the number of pores. The larger the spectral site, the greater the number of pores. The area and value of a single spectral peak positively correlate to the number of pores of the corresponding size [20]. Fig. 7 shows the T2 spectrum characteristics of the limestone samples subjected to different freeze-thaw cycles. Table 2 shows the T2 spectrum parameters of the limestone samples subjected to different freeze-thaw cycles. As seen from Fig. 7 and Table 2, with the increase of the number of freeze-thaw cycles, the T2 spectrum of the limestone sample changes significantly, and the signal strength of the T2 spectrum increases gradually, indicating that the pores of the limestone samples become larger and more prominent with the increase of the number of freeze-thaw cycles. The T2 spectra of both the dry sample and the sample subjected to 5 freeze-thaw cycles show two wave peaks. In addition, the T2 spectrum of the dry sample exhibits poor wave continuity. However, the T2 spectra of most samples subjected to 10, 15, and 20 freeze-thaw cycles show good wave continuity with single wave peaks, which indicates that with the increase of the number of freeze-thaw cycles, the connectivity of the pores and fissures in the limestone samples is gradually enhanced. The area under the T2 spectrum distribution curve indirectly reflects the porosity of the sample. Fig. 7 and Table 2 show that the T2 spectrum peak area of the limestone sample subjected to freeze-thaw cycles is increased compared with that of the dry sample, indicating that the porosity of the limestone sample increases after the freeze-thaw cycle.
Fig. 7 T2 spectra of limestone samples subjected to different freeze-thaw cycles

Tab. 2 T2 spectrum parameters of limestone samples subjected to different freeze-thaw cycles
The impact of freeze-thaw cycle count on the mechanical properties of limestone requires a uniaxial compression test to be carried out on the limestone samples subjected to different numbers of freeze-thaw cycles with the RMT-150C mechanical testing equipment. Again, the influence of freeze-thaw cycles on the elastic modulus, uniaxial compressive strength of the limestone sample is examined. Fig. 8 shows the stress-strain curves of the limestone samples subjected to different freeze-thaw cycles. This figure indicates that, except for the dry samples that had no failure stage after the peak, the stress-strain curves of the 5 kinds of samples subjected to different freeze-thaw cycles went through the initial compaction stage, the linear elastic the plastic yield failure stage. With an increase in the number of freeze-thaw cycles, the failure stage of the stress-strain curve begins to appear after the peak. Beyond 20 freeze-thaw cycles, the stress-strain curves of all the samples show a prominent post-peak failure stage, indicating that the plasticity of the limestone samples is increased after the freeze-thaw cycles.

Table 3 shows the mechanical parameters of the limestone samples subjected to different freeze-thaw cycles. As indicated from Table 3, the limestone sample’s peak compressive strength and elastic modulus are reduced to varying degrees after different freeze-thaw cycles. The average peak compressive strength is 170.75 MPa for the dry samples, 145.61 MPa, 133.26 MPa, 121.67 MPa, and 106.78 MPa for the limestone samples subjected to 5, 10, 15, and 20 cycles, respectively. Compared to the dry samples, the peak compressive strength of the samples after 5, 10, 15, and 20 freeze-thaw cycles is decreased by 14.7%, 22.0%, 28.7%, and 37.5%, respectively. The average elastic modulus is 59.05 GPa for the dry samples, and 54.30, 52.30, 52.14, and 46.38 GPa for the limestone samples subjected to 5, 10, 15, and 20 cycles, respectively. Compared with the dry samples, the elastic modulus decreases by 8.0%, 11.4%, 11.7%, and 21.5%, respectively.

The above observations indicate that the peak compressive strength and elastic modulus had a significant negative correlation with the number of freeze-thaw cycles. This is because, during the freeze-thaw cycle, a large number of microcracks and pores are formed. Consequently, the microscopic mechanical parameters characterizing limestone are reduced due to the existence of the cracks.
Fig. 8 Stress-strain curves of limestone samples subjected to different freeze-thaw cycles

Tab. 3 Mechanical parameters of limestone samples subjected to different freeze-thaw cycles

| No. | Number of freeze-thaw cycles | Compressive strength/MPa | Average compressive strength/MPa | Elastic modulus/GPa | Average elasticity modulus/GPa |
|-----|-----------------------------|--------------------------|----------------------------------|--------------------|-------------------------------|
| A-1 | 0                           | 183.82                   | 165.87                           | 146.94              | 135.77                        |
| A-2 | 0                           | 165.66                   | 151.88                           | 138.01              | 121.07                        |
| A-3 | 0                           | 162.78                   | 151.88                           | 138.01              | 121.07                        |
| B-1 | 5                           | 146.94                   | 151.88                           | 138.01              | 121.07                        |
| B-2 | 5                           | 165.66                   | 151.88                           | 138.01              | 121.07                        |
| B-3 | 5                           | 162.78                   | 151.88                           | 138.01              | 121.07                        |
| C-1 | 10                          | 137.91                   | 126.09                           | 121.07              | 121.07                        |
| C-2 | 10                          | 137.91                   | 126.09                           | 121.07              | 121.07                        |
| C-3 | 10                          | 137.91                   | 126.09                           | 121.07              | 121.07                        |
| D-1 | 15                          | 121.07                   | 121.07                           | 121.07              | 121.07                        |
| D-2 | 15                          | 121.07                   | 121.07                           | 121.07              | 121.07                        |
| D-3 | 15                          | 121.07                   | 121.07                           | 121.07              | 121.07                        |
| E-1 | 20                          | 102.82                   | 102.82                           | 102.82              | 102.82                        |
| E-2 | 20                          | 106.78                   | 106.78                           | 106.78              | 106.78                        |
| E-3 | 20                          | 101.74                   | 101.74                           | 101.74              | 101.74                        |

5 Conclusions

In using the low-field NMR test and the uniaxial compression tests, the impact of freeze-thaw cycles on the microstructure and mechanical properties, composition characteristics, stress-strain curve, compressive strength, and elastic modulus of limestone subjected to specific cycle counts were analyzed, and the following conclusions were drawn:

(1) The main components of the limestone sample are calcium carbonate, iron oxide (FeOx), feldspar, and
quartz. The number of freeze-thaw cycles has a significant effect on the microstructure of limestone. Compared with the dry samples, numerous new cracks appeared during the freeze-thaw cycle, and the crack width increases with the number of freeze-thaw cycles.

(2) The number of freeze-thaw cycles has a significant effect on the pore structure characteristics of limestone. With an increase in the number of freeze-thaw cycles, the signal intensity of the T2 spectrum tends to increase gradually. As a result, the peak in the T2 spectrum of the limestone samples gradually becomes a single peak, and the connectivity of the internal pores is steadily enhanced.

(3) The number of freeze-thaw cycles significantly affects the peak compressive strength and elastic modulus of limestone. Compared with that of the dry limestone samples, the peak compressive strength of the limestone samples subjected to 5, 10, 15, and 20 freeze-thaw cycles decreased by 14.7%, 22.0%, 28.7%, and 37.5%, respectively. The elastic modulus also reduced by 8.0%, 11.4%, 11.7%, and 21.5%, respectively.

Conflict of Interests

The authors declare that there is no conflict of interest regarding the publication of this paper.

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