

Study on time-effect to the strength deterioration of carbonaceous mudstone under water-rock interaction

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ABSTRACT

While excavation activities in mountain terrain regions, the exposure of carbonaceous mudstone could deteriorate the strength under the water-rock interaction, which impacts slope stability and affects the safety of the engineering project. The present study conducted the Point Loading Test (PLT), Acoustic Emission (AE), and Scanning Electron Microscope (SEM) to investigate the degradation of its mechanical properties under the water-rock interaction. The results reveal that: (1) The Total Organic Carbon (TOC) content and water content correspond to a negative correlation, and the carbonaceous mudstone possesses a softening boundary which makes the mudstone strength decay rapidly resulting in the strength decrease by 80% once the water content exceeds the boundary value; (2) Water-rock interaction weakens the acoustic emission capacity and the acoustic emission characteristic in the failure process of carbonaceous mudstone with different TOC can reflect the influence of water-rock interaction on its macroscopic strength and fracture structure deterioration, and the damage degree under immersion is over 50%; (3) The inhomogeneity of water-rock interaction causes unbalanced forces in the rock, as dissolved soluble minerals and oxidation-reduction of organic matter accelerate the deterioration of carbonaceous mudstone making the water-rock interaction is slowed down by the water-swelling filling of minerals in a time period. Therefore, this study has investigated the strength deterioration of carbonaceous mudstone subjected to water-rock interaction from macro-scale and micro-scale perspectives, which provides a reference for preventing and controlling carbonaceous mudstone deterioration resulting from catastrophes.

Keywords: Carbonaceous mudstone; Point loading test; Water-rock interaction; Acoustic emission; Scanning Electron Microscope.

1. INTRODUCTION

Carbonaceous rock distributes in Guangxi province widely. It contains organic carbon which normally coloured in black or dark-grey and classified into carbonaceous mudstone, shale, and limestone [1]. In a micro investigation, carbonaceous mudstone possesses numeral pores which makes it intruded by water easily and disintegrated under water actions. In recent year, new highway construction is booming in mountain region in Guangxi [2]. After excavating terrain during engineering activities, the exposure of carbonaceous rock is easy to absorb water and softens under the intrusion of rainfall, disintegrating rocks into debris as a consequence. Furthermore, the intensification of water-rock action can lead to the marginalization of carbonaceous rocks, which influences slope stability in such carbonaceous mudstone area. Therefore, study the water-rock softening effect on carbonaceous mudstone is practically significance on monitoring slope stability and preventing landslide catastrophe.

Currently, scholars domestic and worldwide have studied the mechanical properties subjected to water-rock interactions. ZHANG *et al.* [3] concluded a short review in laboratory tests, which focuses on the water weakening of rock strength and its influencing factors including water content, immersion time, and wetting-drying cycles. In considering water content as influencing factor on rock strength, NOËL *et al.* [4] proposed that the plasticity of rock mass can be increase with increasing of water content while brittleness is reduced.

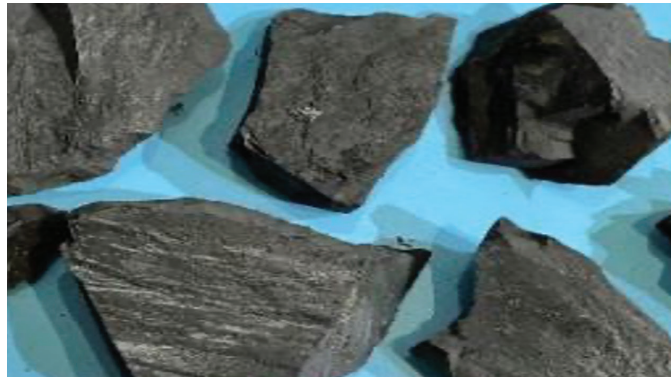


Figure 1: Carbonaceous rock specimen.

Moreover, ZHU *et al.* [5] and MA *et al.* [6] concluded from experiments that rock mechanical parameters become weakened as the immersion time increases. Since many engineering activities are involving wetting–drying cycles, for instance the rocks in exposed slopes, the water-drying cycle treatment has a significant impact on the mechanical and physical properties [7–9]. Moreover, GUO and HOU [10] conducted laboratory tests on the carbonaceous phyllite in southern mines and the results show that the carbonaceous phyllite belongs to moderately expansive soft rock which possesses strong water absorption resulting to soften and disintegrate easily. LIU *et al.* [11] carried out real-time monitoring of the instability characteristics such as displacement and seepage pressure of the tunnel surrounding rock in Guangxi and studied the monitoring values by using Singh-Mithel model to analyse the failure creep characteristics of the carbonaceous rock. LIU *et al.* [12] analysed the compressive and shear strength of carbonaceous mudstone at different times of immersion and concluded that the strength of carbonaceous mudstone decreased sharply with the increase of immersion time. ZENG *et al.* [13] utilized the CT triaxial test to study the meso-stress deformation and damage evolution of carbonaceous mudstone providing a new method for further analysing the physical and mechanical properties of carbonaceous mudstone. DAI [14] carried out laboratory tests based on the softening mechanism of weak intercalated carbon mudstone samples and verified that the creep characteristics of weak intercalated carbon mudstone influencing slope stability. DHAKAL *et al.* [15] revealed the intrinsic relationship between mineralogical characteristics and disintegration resistance of sedimentary rocks through experiments. ZHOU *et al.* [16] studied the degradation characteristics of cohesion and internal friction angle of red sandstone, mudstone, and carbonaceous mudstone under the water-rock interaction and established a finite element model for slope stability analysis. ZHAO *et al.* [17] deduced the composite damage variable considering the coupling of macroscopic as well as microscopic defects and verified the rationality of the model by citing the uniaxial compression test data of rock. WANG *et al.* [18] studied the law of microcracks in rock samples caused by the water-rock effect from limestone and mudstone by X-Ray Diffraction (XRD), Total Organic Carbon (TOC) content, pyrolysis, thin petrography, Scanning Electron Microscope (SEM). MA *et al.* [19] researched the hydraulic and mechanical properties of CPB samples under wetting-drying cycles by conducting permeability tests, nuclear magnetic resonance (NMR) tests and uniaxial compression tests. ZHOU *et al.* [20] considered the weakening effect of red sandstone under the action of the Three Gorges water level change and studied the microscopic changes of rock samples through Scanning Electron Microscope (SEM). Under the action of water immersion with the adding of external loadings, the cracks and voids of carbonaceous mudstone have been gradually developed, and the porosity evolution model is obtained by fitting experimental results [21–23].

Considering carbonaceous mudstone is a kind of water-sensitive and easily disintegrated material, this presented study conducted point loading test and acoustic emission test on collected specimen which immersed by water in a series time and utilized Scanning Electron Microscope (SEM) to analyze strength deterioration of carbonaceous mudstone subjected to water-rock interactions.

2. SPECIMEN PREPARATION AND EXPERIMENT SET-UP

2.1. Preparation of carbonaceous rock specimens

The carbonaceous mudstone specimens were collected on-site from the slope of K42+200 in the Bama-Pingguo Expressway link in Guangxi and the testing specimens were prepared based on the National Standard for Engineering Rock Test Methods. Large rock samples were cut into test blocks controlling the quality of each

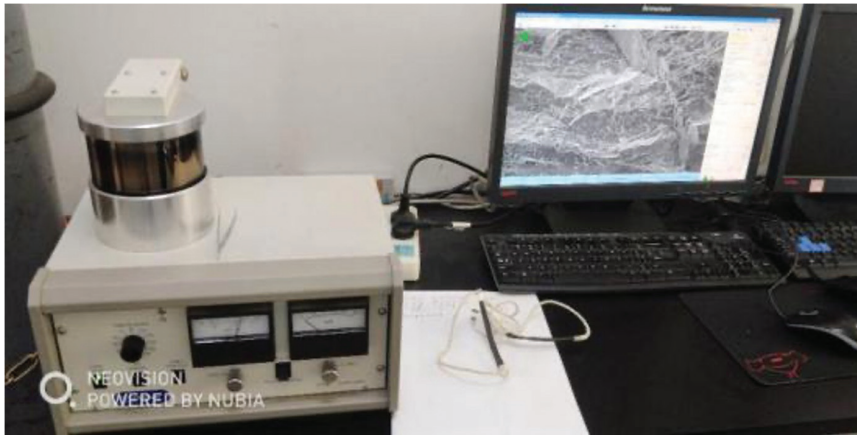


Figure 2: Scanning test on carbonaceous mudstone inspected by FEI Quanta 250 electron scanning system.

Table 1: Mass and atomic component percentage of carbonaceous mudstone specimens (%).

ELEMENT	MASS PERCENTAGE/%	ATOMIC COMPONENT PERCENTAGE/%
C	18.479~33.728	28.358~46.226
O	21.188~40.539	34.442~42.327
Si	2.031~25.001	1.206~16.487
Al	6.837~16.640	5.402~11.418
Ca	1.232~25.201	0.563~10.641
N	0.514~2.212	0.667~2.208
K	0~3.797	0~1.788
Na	0.211~1.128	0.151~0.907
Fe	0.021~2.212	0~0.723

sample ranging from 40g to 60g, see Figure 1. Furthermore, the main component of the carbonaceous mudstone, the FEI Quanta 250 electron scanning system was utilized to scan and analyze the initial composition of the carbonaceous mudstone specimens, see Figure 2.

Under the investigation by an electron scanning system, carbonaceous mudstone is mainly composed of quartz, calcite, marble, illite, kaolinite, and montmorillonite, with Total Organic Carbon (TOC) content in a range from 0.988% to 6.706%. FEI Quanta 250 environmental electronic scanning X-Ray Diffraction (XRD) analysis shows that the composition elements of rock samples are shown in Table 1. From a micro-scale point of view, chemical elements such as Carbon, Oxygen, Silicon, et al.

2.2. Test scheme

In the present experiment, the carbonaceous rock specimens with TOC content of 0.998%, 2.456%, and 6.706% were selected for further water-rock interaction tests, and the test scheme is shown in Table 2. Furthermore, the points loading test was carried out for testing, with the utilization of the STDZ-3 Rock Point Loading Tester, see Figure 3. Based on the code proposed in the industry standard, the Test Methods of Rock for Highway Engineering (JTGE41-2005), the number of rock core specimens in the same water content state is taken to point load testing, and 5 to 10 rock cores are taken from each group. Thus, in this study, 10 samples are taken from each group for repeated testing.

This tester consists of a loading system and a pressure sensor, with the maximum pressure emitting from the sensor at 100KN. Furthermore, a PCI-2 acoustic emission monitor was applied to monitor the acoustic emission signals emitting from specimens with different TOC content and monitor the failure process. Setting sensors above the specimen surface with gel coated evenly on the specimen surface as an acoustic transmission medium, see Figure 4.

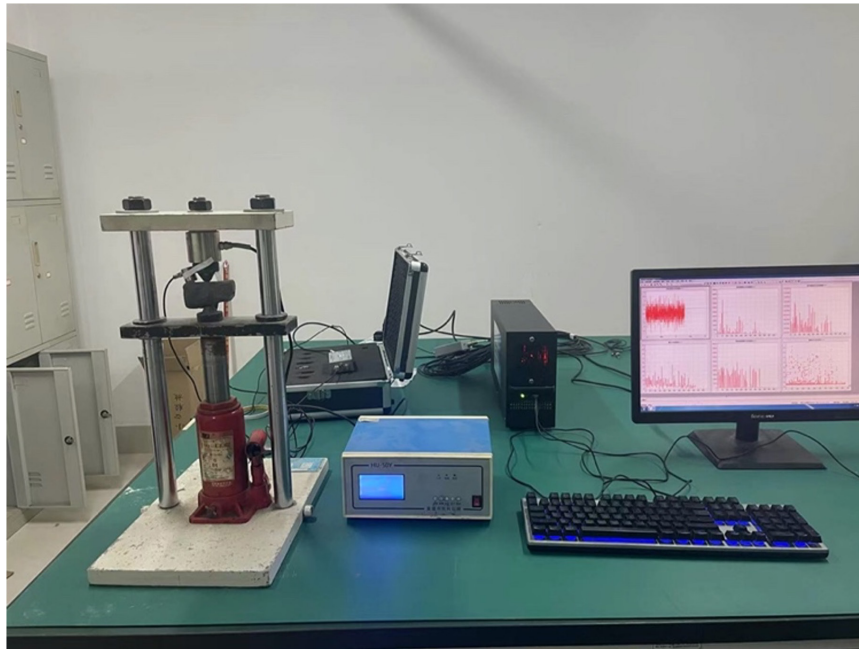


Figure 3: STDZ-3 Rock Point Loading Tester.

Table 2: Testing scheme.

TOTAL ORGANIC CARBON (TOC) CONTENT	IMMERSION TIME/D	TEST ITEM
0.998% 2.456% 6.706%	0	PLT; AE; SEM
	0.5	PLT; AE; SEM
	1	PLT; AE; SEM
	2	PLT; AE; SEM
	5	PLT; AE; SEM
	10	PLT; AE; SEM
	15	PLT; AE; SEM
	30	PLT; AE; SEM

*PLT: Point Loading Test; AE: Acoustic Emission; SEM: Scanning Electron Microscopy.

3. SOFTENING EFFECT ANALYSIS

3.1. Water absorption characteristics of carbonaceous mudstone in different TOC content

To study the water absorption characteristics of carbonaceous mudstones in different Total Organic Carbon (TOC) content, the initial condition is set by putting specimens into an oven for evaporation till a content mass.

According to the industry standard, the Test Methods of Rock for Highway Engineering (JTGE41-2005), the temperature for drying is set as 105 degrees. Afterward, carbonaceous rocks with different TOC content are soaked in water for a period of 0, 0.5, 1, 2, 5, 10, 15, and 30 days respectively. At the same time, the water content of the carbonaceous mudstone at different stages during corresponding the time is measured and numerical values are plotted in Figure 3.

It can be seen from Figure 5 that the moisture content of carbonaceous mudstone is significantly positively correlated with TOC content and soaking time. The water absorption rate of carbonaceous mudstone in the initial stage is increasing rapidly in the first quatre stage during the time while the specimen is gradually saturated in the later stage and the water content tends to be flatted. Moreover, the maximum water absorption rate and water absorption rate of carbonaceous mudstone are positively correlated with TOC content. Elaborately, the higher the TOC content is, the faster the water content rises in a short time, the later the inflection point of



Figure 4: Point Loading test with caustic signals collections.

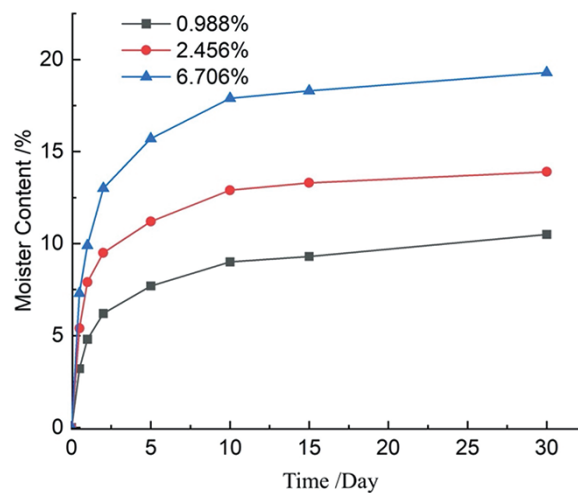


Figure 5: Moisture content of carbonaceous mudstone in different TOC content.

water absorption appears, and the stronger the water absorption of carbonaceous rocks. With the increase in soaking time, the water absorption rate increases sharply at the first stage, and the trend increases slowly starting from the 5-day. The saturated water absorption corresponding to TOC content of 0.988%, 2.456%, and 6.706% is 10.501%, 13.902%, and 19.312%, respectively. Therefore, the water absorption characteristics of carbonaceous rocks are affected by the initial water content and TOC content.

3.2. The strength variation in different TOC content under water-rock interactions

To study the deteriorated effect under the water-rock interaction, the point loading test of carbonaceous mudstone with different TOC content in a soaking period is carried out. The change of yielding strength with the increase in soaking time is obtained as plotted in Figure 6.

As Figure 6 presented, it reveals that the yielding strength of carbonaceous mudstone shows an obvious attenuation trend with the increase of soaking time, in which the rate of strength declines rapidly before 5 days and tends to be flat in the following days. For carbonaceous mudstone with a TOC content of 0.998%, the yielding strength is 75.000MPa in the initial stage and declines to 24.600MPa after soaking for 30 days, with the decline rate being 67.221%. Similarly, the yielding strength of the specimen with a TOC content of 2.456% is 67.590MPa before being immersed and reaches to 9.300MPa after being immersed in water for 30 days, with

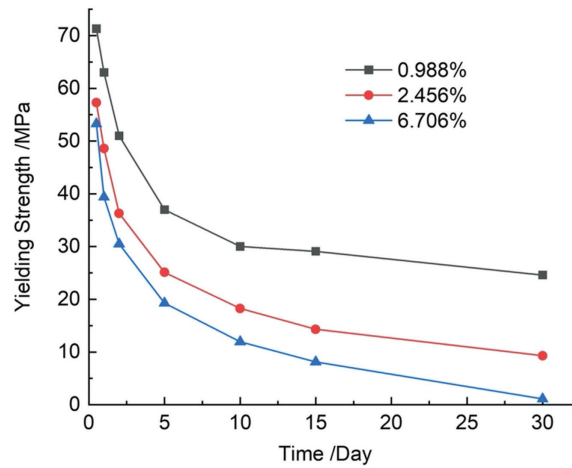


Figure 6: Yielding strength after soaking with different TOC content.

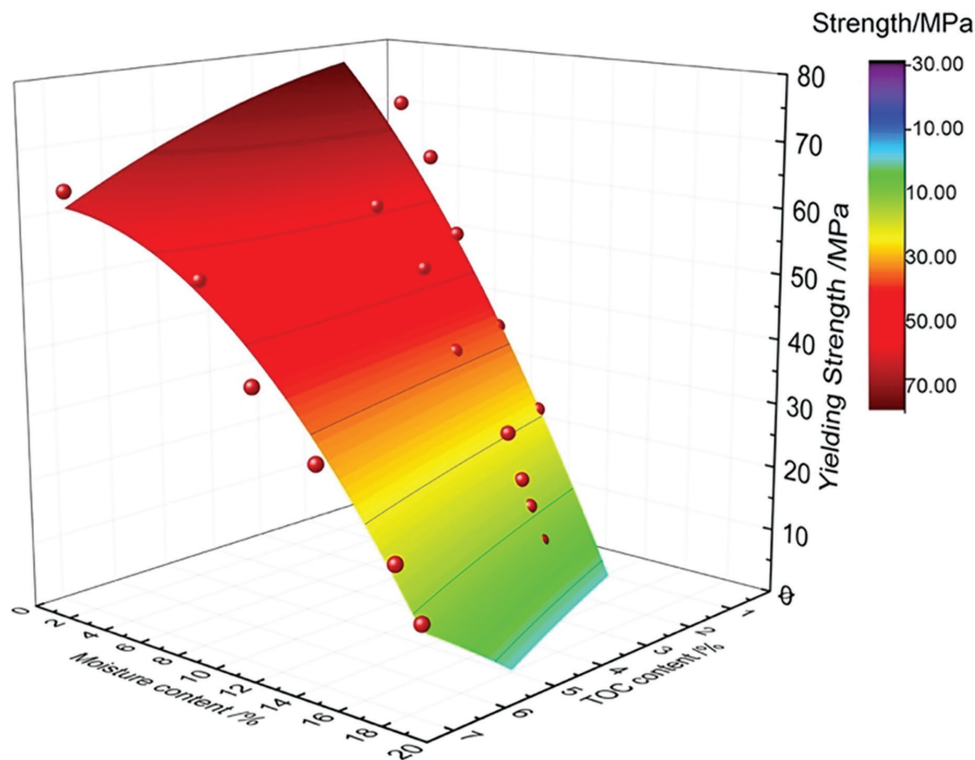


Figure 7: Fitting diagram of strength prediction of carbonaceous mudstone considering affecting parameters including TOC content, moisture content, and yielding strength.

a strength reduction rate of 86.240%. While the specimen which TOC content of 6.706% is 62.880MPa and 1.100MPa for soaking in water for 0 and 30 days respectively, with a decrease rate of the strength of 98.250%.

It can be observed that the rapid strength deterioration of carbonaceous mudstone mainly occurs within 10 days. When the soaking time is within 10 days, the point load strength decreases obviously with the increase of soaking time. Specimens in different TOC contents possess different water absorption capabilities, saturated water content, and strength characteristics of rocks. Moreover, it can be calculated that specimen of 0.988%, 2.456%, and 6.706% TOC content possesses softening boundary which evaluated by water absorption capability corresponding to 8.403%, 12.241%, and 16.710%, respectively. When the water content is lower than the softening boundary value, the carbonaceous mudstone is sensitive to water absorption. The water content has a great influence on the strength. When the water content exceeds the softening boundary value, it is insensitive to water absorption and softening, resulting strength deteriorated by more than 80% of the total strength.

3.3. Strength prediction and analysis during the water-rock interaction

According to analysis in the previous section, it appears that TOC content, water content, and yielding strength are related to the deterioration rate of mudstone strength. Considering those affecting parameters, the strength prediction surface obtained by the fitting function is illustrated in Figure 7 which predicts the strength of carbonaceous mudstone subjected to water-rock interactions. It reflects that the strength shows a negative correlation with TOC content as well as water content, with a correlation coefficient of $R^2 = 0.972$. The strength prediction function with affecting parameters is illustrated in Equation 1.

$$P = 78.25 - 0.864\omega - 3.983T - 0.269\omega^2 - 0.143Y^2 + 0.972\omega T \quad (1)$$

where, P means strength prediction of carbonaceous mudstone; Y notes the yielding strength obtained by loading tests, ω presents moisture content, and T is TOC content.

4. ACOUSTIC EMISSION AND DETERIORATION ASSESSMENT OF CARBONACEOUS MUDSTONE

4.1. Analysis of acoustic emission capacity and accumulative energy to carbonaceous mudstone

To investigate the relationship between carbonaceous mudstone damaging conditions from the macro-mechanical perspective, the Acoustic Emission (AE) tests are conducted, and the numerical statistics on acoustic emission capacity and acoustic emission accumulative energy corresponding to different TOC contents in water immersion are illustrated in Figure 8 and Figure 9 respectively.

There are some observations that can be concluded that before immersing specimens into the water, the initial acoustic emission capacity with TOC contents in 0.988%, 2.456%, and 6.706% are 11.500×10^6 , 10.520×10^6 , 9.920×10^6 respectively, while the acoustic capacity declines to 4.820×10^6 , 10.520×10^6 , and 1.900×10^6 , with the corresponding declination rate, is 58.089%, 68.630%, and 80.839%. Intuitively, TOC content affects its acoustic emission capacity, a higher TOC content with a relatively lower acoustic emission capacity. Furthermore, the acoustic emission capacity reflects internal fissures developed in the carbonaceous mudstone sample, reflecting its internal damage condition to some degree. From Figure 8 above, it can be seen that the internal damage of the carbonaceous mudstone is related to the TOC content and water content. Thus, as the time of water immersion moves on, the intrusion of water makes minerals in the carbonaceous mudstone soften and makes the acoustic emission capacity decline consequently.

From a different point of view, Figure 9 plots the results of acoustic emission cumulative energy under immersion time with different TOC content. When TOC content is 0.988%, the cumulative energy of acoustic emission is $4163.110\mu\text{J}$ within 0-day of immersion and it becomes $2245.420\mu\text{J}$ after 30-day of immersion, with a reduced rate of 46.062%. For specimens with a TOC content of 2.456%, the cumulative energy of acoustic emission is $3815.120\mu\text{J}$ after soaked for 30 days and the cumulative capacity of acoustic emission is $1948.750\mu\text{J}$, with a reduced rate of 48.920%. Considering the TOC content of 6.706%, the cumulative energy of acoustic emission is $3345.130\mu\text{J}$ initially and reduce to $1588.370\mu\text{J}$ after 30-day soaking in water, with a reduction rate was 52.509%. With the increase of immersion time, the energy stored in the specimen is lower, and the

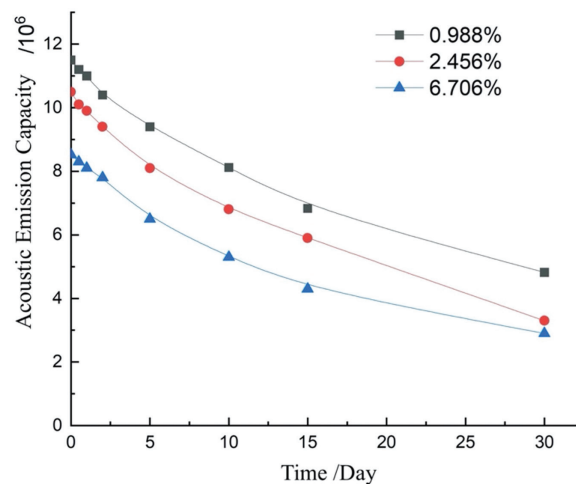


Figure 8: Acoustic emissions capacity subjected to water-rock interaction with different TOC content.

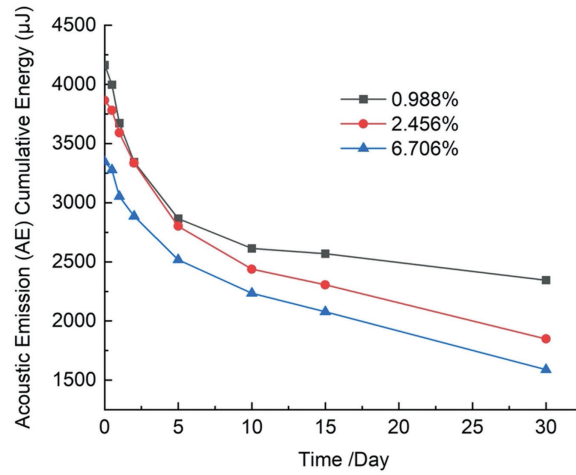


Figure 9: Acoustic emission accumulative energy under water-rock interaction with different TOC content.

Table 3: Damage condition of carbonaceous mudstone under water-rock interaction

TOC DAMAGE CONDITION TIME	0.988%	2.456%	6.706%
0	0	0	0
0.5	0.033	0.041	0.018
1	0.081	0.081	0.034
2	0.146	0.114	0.059
5	0.244	0.252	0.164
10	0.340	0.358	0.262
15	0.445	0.463	0.343
30	0.608	0.594	0.538

accumulated energy of acoustic emission decreases. Meanwhile, the higher the TOC content, the weaker the cumulative ability of acoustic emission. In the process of soaking and softening, the TOC content has a significant impact on acoustic emission, and the higher the TOC content, the greater the decline of acoustic emission.

4.2. The deterioration assessment under water-rock interaction

As mentioned previously, the strength of carbonaceous mudstone is significantly affected by its moisture content, TOC contents, and yielding strength. This section intends to assess the internal damage condition of mudstone samples under the water-rock interaction by conducting acoustic emission detection research. Moreover, TANG and XU [24] proposed a method to convert the rock damage coefficient through the cumulative number of acoustic emissions with its corresponding certain time. Thus, noting D as the damage condition of the specimen under water-rock interaction which is assessed by Equation 2, presenting the results in Table 3 and Figure 10.

$$D = \frac{N_t}{N_m} \tag{2}$$

where: N_t notes as acoustic emission capacity to a certain immersion time; N_m presents initial acoustic emission cumulative number.

Figure 8 illustrates that with the increase in soaking time, the damage condition of the rock becomes severe. After 30 days of specimen immersion, the damage condition reaches 60.802%, 59.350%, and 53.820% which corresponds to the TOC content of 0.988%, 2.456%, and 6.706% respectively. Therefore, after the excavation of mountain terrain, making carbonaceous mudstone exposed to the air, the intrusion of water making mudstone immersed in a short period of time will cause rapid deterioration to carbonaceous mudstone. The surface should be sealed as soon as possible after excavation to avoid contact between the rock surface and water in reducing the damage to the carbonaceous mudstone avoiding slope collapses and slides along the bedding instability.

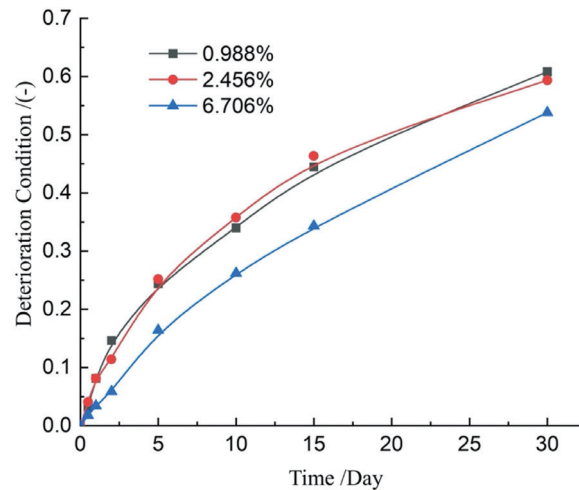


Figure 10: Deterioration condition of specimens in immersion time with different TOC content.

5. SCANNING ELECTRON MICROSCOPIC (SEM) INVESTIGATION OF CARBONACEOUS MUDSTONE DETERIORATION

Carbonaceous mudstone has special hydraulic properties. Although macro perspectives can reflect its mechanical properties, it has certain limitations. This section from the micro point of view analyses the deterioration difference between weathering and water-rock interaction processes and reveals its micro-deterioration characteristics. Taking the excavation specimen exposed on 0-day, 10-day, and 30-day weathering conditions and the specimens subjected to water-rock interaction with corresponding time, scan the specimens in different conditions under SEM magnifying 200 times, 1000 times, and 3000 times to capture its condition in a micro scale, see Figure 11 and Figure 12.

5.1. Carbonaceous rock under weathering conditions

It can be seen in Figure 11a that the surface of carbonaceous mudstone is mostly distributed in stripes, mixed with organic minerals, and there are a few clays mineral crystals scattered in the gaps. The clay mineral crystals exist in flakes and formed a structure. Furthermore, the flakes are connected face-to-face, and neatly arranged, with relatively low pores and high density. Although the thickness of the crystal flakes is basically the same, the length shows different, which results in a large number of voids on the carbonaceous mudstone surface. Besides, Figure 11b shows that after 10 days of natural weathering, the surface of the structure is rough, and there are occasional pores among the clusters. Moreover, the clay mineral crystals are evenly distributed, and the crystals are mostly scale-like. Carbonaceous mudstone mineral crystals exist in thin slices with poor structure, and cracks are often seen in the thin slices. The flakes are connected face-to-face, and neatly arranged, with relatively low pores and high density. There are some independent clay mineral grains scattered on the surface of the mudstone. Additionally, it can be seen from Figure 11c that the overall surface of mudstone is rough, and the clay mineral crystals show the properties of swelling, dispersion, and opening-like petals. The crystals and crystals are overlapped into crystal clusters and the arrangement of the crystal clusters is uneven, making many gaps between the clusters and revealing structure condition is poor. Thus, under natural weathering, changes in temperature and humidity cause the deterioration of the carbonaceous mudstone surface, which leads to further aggravated under-water-rock interaction with carbonaceous mudstone.

5.2. Carbonaceous rock subjected to water-rock interaction

As Figure 12 presents that the clay minerals in the pore-water-rock interaction led to rock mass softening and expansion. With the increase of contact area under water-rock interaction, the softening of carbonaceous mudstone accelerated while the softening and swelling of medium clay minerals intensified. After carbonaceous mudstone is immersed in water, clay minerals that are soluble in water successively transfer to the rock surface. At the same time, due to the variability of the rock and the uneven distribution of microscopic components, the number of crystal aggregates on the rock surface. The soluble minerals in the carbonaceous mudstone undergo dissolution, dissolving in water and causing quality loss, changing the rock structure, and making the internal structure of the rock loosen when transported to the rock surface. In the development of fissures, the mineral water swells and fills the voids, preventing the water from further deepening. Hence, the disintegration of

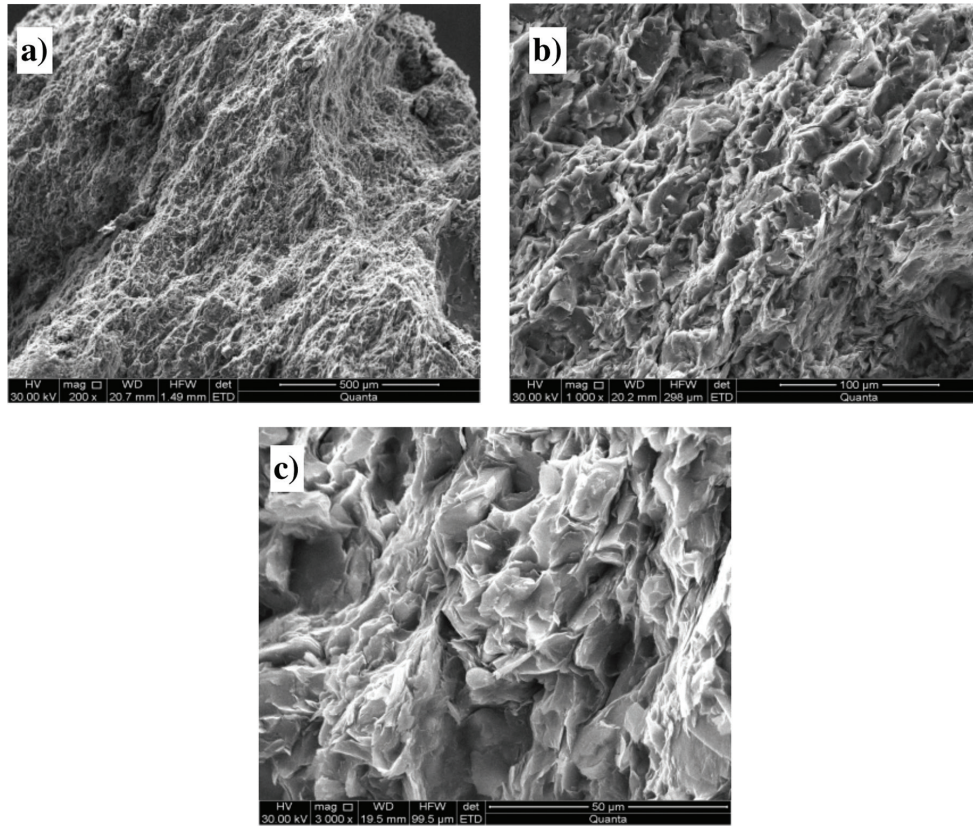


Figure 11: SEM micro-scale investigation under different weathering time.

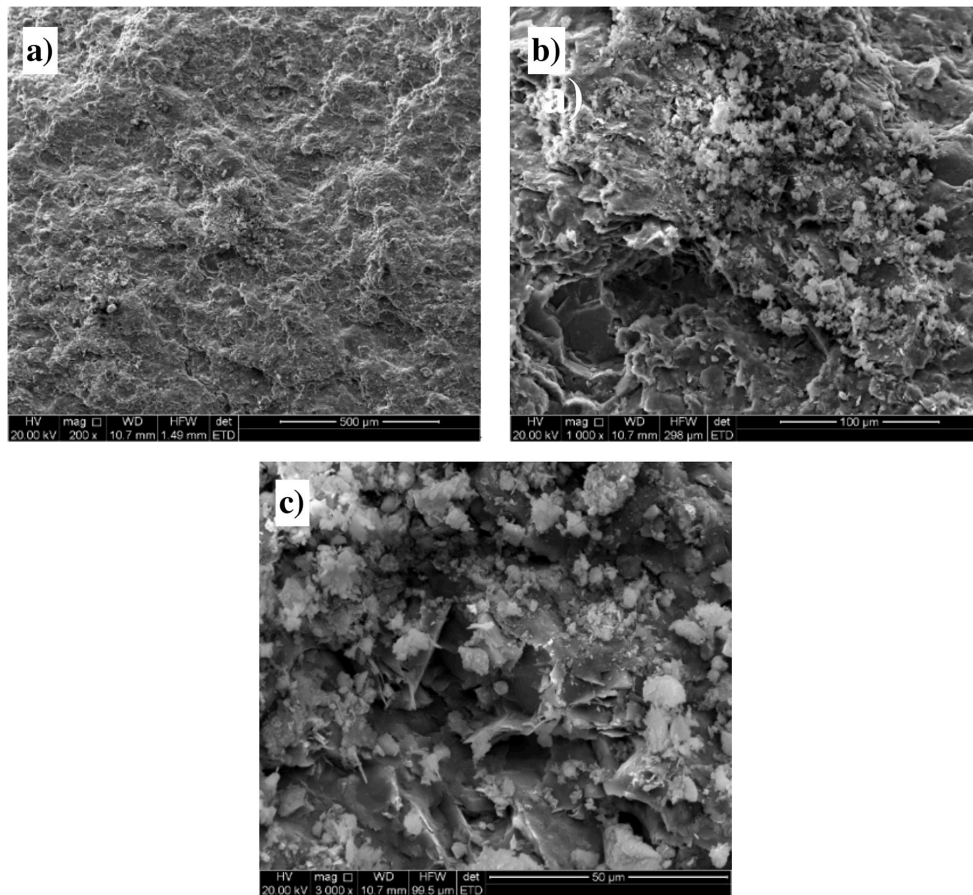


Figure 12: SEM micro-scale investigation under different immersion time.

carbonaceous rock is mostly a process of gradual disintegration from shallow to deep. The results and the acoustic emission results mutually confirm that the carbonaceous mudstone deteriorates and the acoustic emission energy decreases after the mineral absorbs water, making the energy consumed. Thus, during the construction, to avoid the deepening of disintegration and the intensified action of water and rock, attention should be paid to the prevention and treatment of the development of surface cracks.

6. CONCLUSION

In this study, the strength deterioration and damage condition assessment of carbonaceous mudstone are studied by conducting a series of tests including the Point Loading Test (PLT) and Acoustic Emission (AE). Furthermore, the micro-scale investigation which examines by Scanning Electron Microscope (SEM) inspecting natural weathering and water immersion of carbonaceous mudstone has been performed. The conclusions are summarized as follows:

- (1) The water absorption rate of carbonaceous mudstone starting from the initial dry state is rapid, while the water absorption rate decreases significantly with the increase of rock water content. The higher the TOC content, the higher the saturated water absorption rate. The 10-day reaches to the boundary of water absorption, and the water absorption rate decreases significantly, with the saturated moisture contenting water between 10.5% and 19.3%.
- (2) TOC content has a negative correlation with the point loading yielding strength of carbonaceous mudstone. Carbonaceous mudstone with different TOC content possesses boundary conditions for a strong softening effect after water absorption. The boundary condition in values is 10.501%, 13.902%, and 19.312% respectively corresponding to TOC content of 0.988%, 2.456%, and 6.706%. When the water content exceeds to the boundary condition, the softening effect of water and rock becomes severe. Furthermore, the strength prediction formula obtained by multi-parameter fitting can effectively deduce the strength attenuation law of carbonaceous mudstone and provide a reference for the prevention and reinforcement of carbonaceous mudstone slopes.
- (3) Under the water-rock interaction, acoustic emission energy is absorbed by the material, making the acoustic emission capacity and accumulative energy decrease. TOC content of carbonaceous mudstone impacts its acoustic emission capacity revealing that a higher TOC content processes a relatively lower acoustic emission capacity. The longer the immersion time of carbonaceous mudstone, the intrusion of water making the materials softening and leading to deterioration of acoustic emission capacity. Additionally, the assessment of the damage condition exceeds more than 50% of internal damaging. Therefore, the surface deterioration treatment measures should be taken as soon as possible after the excavation of the carbonaceous rock slope.
- (4) Through performing Scanning Electron Microscope (SEM) investigation on analyzing the deterioration of carbonaceous mudstone, the water-rock interaction causes the changes of carbonaceous mudstone microstructure, causing unbalanced forces between rocks. Furthermore, the dissolved soluble minerals and reduction of oxidation in organic matter have relatively accelerated the material strength deterioration under the water-rock interaction comparing with the natural weathering conditions.

7. ACKNOWLEDGMENTS

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