Influence of Drying–Wetting Cycle of Acid Solution on the Mechanical Properties of Sandstone

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Abstract. Ensuring the stability of reservoir slopes requires a comprehensive understanding of the mechanical properties of rocks after the drying–wetting cycles under acidic conditions. In this work, sandstone samples were selected and subjected to cyclic drying–wetting treatments with aqueous solutions of pH 7.0, 6.0, 4.0, and 2.0. The uniaxial compression tests and P-wave velocity measurement were carried out to evaluate the variations in the physico-mechanical properties of sandstones with different treatments. The results revealed that the internal structure of sandstones is softened by the drying–wetting cycles, which deteriorate their physico-mechanical properties. As the acidity of the aqueous solution increases, porosity and Poisson’s ratio show an increasing trend, while the strength characteristics, Young’s modulus, and P-wave velocity of sandstones present a decreasing trend. The physico-mechanical properties of sandstones significantly deteriorate from neutral to weakly acidic solutions; however, the deterioration becomes slower as the acidity continues to increase. Moreover, the physico-mechanical properties of sandstones are markedly reduced under strong acidic conditions.

1. Introduction

Due to the alternate fluctuations of the water level of the reservoir, the rock mass on the bank slope has been in the "saturated-dried" drying–wetting cycle during the long-term operation of the reservoir. This repeated water–rock interaction will cause non-negligible fatigue damage to the mechanical properties of slope rock mass, which tends to be much stronger than the continuous degradation of the rock mass due to continuous immersion [1]. Without effective prevention and treatment, long-term drying–wetting cycles may cause geological disasters, such as mudslides, collapse, and landslides, thereby threatening the stability and safety of the reservoir area. At present, many researchers have carried out a large number of experimental studies on this topic. For example, Yao et al. [3] found that the drying–wetting cycle enhanced the ductility of sandstones, although it has a weakening effect on the compressive strength, elastic modulus, cohesion, and internal friction angle of sandstones. Liu et al. [4] used the scanning electron microscopy (SEM) and discrete element numerical simulation in order to analyze from a microscopic perspective the degradation mechanism of the mechanical properties of argillaceous sandstones under different drying–wetting cycles. Bin et al. [5] studied the effects of drying–wetting cycle and loading rate on the dynamic tensile properties of red sandstone, and found that the dynamic tensile strength was negatively correlated with the number of dry–wet cycles. They further reported that each drying–wetting cycle conditions was influenced by the presence of a critical
loading rate, such that the tensile strength of sandstone remained substantially unchanged when the rate exceeded the threshold loading.

In addition, water in nature is not completely neutral and mostly consists of acidic or alkaline water chemical solution [6]. Therefore, chemical coupling also has an effect on the water–rock interaction process. China is still one of the three major acid rain regions in the world, and in the past years, acid rain disasters have occurred in the Central and Southwestern parts of the country with many large hydropower projects [7]. Under acidic conditions, irreversible chemical reactions occur in the mineral components or cements in rocks, causing the decay and weakening of their physical and mechanical properties. This weakening due to the acidic environment may be more significant than the alternating effects of drying–wetting cycles. Li Ning et al. [8] found that the acidic solution can significantly reduce the mechanical properties of calcareous cemented feldspar sandstones and that the dissolution of the main cement CaCO$_3$ is the main reason for its strength and deformation attenuation. Feng et al. [9] studied the effects of ion concentration and pH on the mechanical properties of granite and found that the degree of reduction of granite strength was affected by H$^+$ concentration. Tan et al. [10] analyzed the intensity change law of marbles and diabase under different types of acids and concentrations. Their results indicated that the deterioration of rock strength depends not only on H$^+$ concentration, but also on acid ion concentration control.

The rock mass of the bank slope in actual engineering practice often suffers from the simultaneous dual effects of the drying–wetting cycle and acid environment. Gaining mastery of the changes in the mechanical properties of the rock mass under this combined action ensures the long-term stability of the bank slope. At present, relatively few studies on rocks under the action of acidity and drying–wetting cycles have been carried out, and the mechanism of rock weakening is still unclear. Thus, in the current study, sandstone samples were subjected to the dry–wet cycle test for 10 times using aqueous solutions of various pH values, and the change law of the physico-mechanical properties of the samples were analyzed to provide reference for the theoretical and applied research in related fields.

2. Materials and methodology

2.1. Specimen preparation

The sandstone specimen selected for this test was taken from Zigui County, Hubei Province. The rock block in its natural state is yellow and has uneven particle size, no obvious joint on the surface, coarse grain structure, and good integrity. Its main mineral components are quartz, feldspar, and clay minerals. All specimens were taken from the same rock block in order to reduce the influence of the non-uniformity of the specimen on the test results. According to the specification requirements, the sandstone specimen is processed into a standard cylindrical shape with a height of 100 mm and a diameter of 50 mm. Within the range allowed by the specification, the flatness and verticality deviation of the two end surfaces of the specimen are strictly controlled.

We used the non-metallic sonic tester to measure the P-wave velocity of each specimen, after which we removed the specimens that significantly deviated from the average value. The screened specimens were divided into five groups of three samples each. Each group was respectively used to determine the basic physical indicators of the porosity, density, and P-wave velocity of the sandstone specimens in the natural state, respectively. The remaining four groups were respectively immersed for 24 hours in four aqueous solutions of pH = 7.0, 6.0, 4.0, and 2.0, which were prepared with distilled water and dilute nitric acid. Then, four groups of rock samples were dried in an electric furnace for 2 hours at a drying temperature of 105°C. Dried rock samples continue to be immersed in the aqueous solutions and underwent drying–wetting cycles for 10 times. The rock specimens soaked in different acid solutions are shown in Figure 1.
2.2. P-Wave velocity measurement
The non-metallic sonic tester RSM-SY5 (T) was used to test the P-wave velocity of the sandstone specimen after the treatment. The RSM-SY5 (T) apparatus has ultrasonic pulse output and input device and can automatically calculate the P-wave velocity of sandstone rock specimens after collecting the signal. During the experiment, the two sensors were placed in the center of the two sides of the rock specimen, after which the surface of the sensor probe was filled with petroleum jelly to make the connection closer. Next, we tested each group of specimens and recorded the average values. The results are shown in Table 1.

2.3. Uniaxial compression tests
The uniaxial compression test was performed on a TAW-3000 rock triaxial servo multi-field coupling testing machine, while the deformation was monitored using an LVDT deformation sensor. Figure 2 presents the specimen installation is shown in. Deformation control is used in the loading process at a control rate of 0.02 mm/min. When the stress is close to the peak value, the loading rate is reduced to 0.005 mm/min until the stress–strain curve falls.

Figure 1. Sandstone specimens soaked in water solutions of pH = 7.0, 6.0, 4.0, and 2.0.

Figure 2. Schematic diagram of the rock specimen installation in uniaxial compression test.
3. Experimental results

3.1. Physical properties
Table 1 presents the P-wave velocity and average density of the rock specimens in their natural state and after undergoing different acidic dry–wet cycles.

| Number of drying–wetting cycles | pH  | Density (g/cm$^3$) | P-wave velocity (m/s) |
|---------------------------------|-----|--------------------|-----------------------|
| 0                               | 7.0 | 2.31               | 3012                  |
| 10                              | 7.0 | 2.30               | 2688                  |
| 10                              | 6.0 | 2.29               | 2427                  |
| 10                              | 4.0 | 2.28               | 2369                  |
| 10                              | 2.0 | 2.26               | 2330                  |

The analysis shows that the densities and P-wave velocities of sandstones under different acidic dry–wet cycles are continuously reduced. The P-wave velocities of the specimens in the natural state and after drying–wetting for 10 times in distilled water (pH = 7.0) are 3012 and 2688 m/s, respectively. Overall, the P-wave velocity of specimens after drying–wetting for 10 times decreased by 10.8% compared to their natural state. This finding indicates that the originally homogeneous and tight sandstone specimens began to produce pores after the drying–wetting cycle treatment; these pores became larger and continued to expand until they formed micro-cracks [11]. The P-wave velocities of the specimens after drying–wetting for 10 times at pH = 6.0, 4.0 and 2.0 solutions are 2427, 2369 and 2330 m/s, respectively. Compared to sandstone specimens treated with pH = 7.0, the P-wave velocities are reduced by 9.0%, 11.2%, and 12.7%, respectively. From neutral solutions to weakly acidic solutions, the P-wave velocities of the samples decreased significantly. This is a chemical reaction of the cement in the sandstone, which leads to the formation of more micro-cracks in the internal structure of the sandstone. Along with weakly acidic solution to strong acidic solution, the P-wave velocities of the sandstone samples slowly decrease.

![Figure 3](image-url)  
**Figure 3.** Relationship between the compressional wave velocity and pH value of sandstone.

3.2. Stress–strain behavior
The stress–strain curve of the whole process can be divided into five stages according to the progressive fracture theory of rock: crack closure section, elastic section, crack stable extension...
section, crack unstable extension section, and post-peak failure section [12]. Figure 4 presents the stress–strain curves of sandstones with different solutions and drying–wetting cycles under uniaxial compression. The sandstones have similar deformation laws under different drying–wetting cycles, but with the increase of acidity, the uniaxial compressive strength of the rock samples decreases significantly, the ductility is relatively enhanced, and brittleness is relatively weakened. This is an acidic corrosion that causes the sandstone mineral particles to dissolve, which makes the sandstone’s porosity larger and leads to obvious nonlinear characteristics during the crack closure stage. The stress–strain curve shows a concave characteristic due to the compression of the initial cracks and voids in the rock.

![Stress-strain curve](image)

**Figure 4.** Sandstone specimen uniaxial stress–strain curve.

### 3.3. Rock strength properties

Young’s Table 2 shows the mechanical parameters of the rock specimens after 10 drying–wetting cycles of different solutions. Figure 5 shows the relationships between the peak strengths and the pH values of specimens with different solutions during the drying–wetting treatment. As can be seen, the peak strength of sandstone decreases when the pH value is reduced. For example, the peak strength of sandstone after pH = 2.0 solution treatment is 69.6% of the overall peak strength at pH = 7.0. The peak strength shows a steep-slow-steep trend with a decrease of pH value. This is due to the chemical reaction of potassium feldspar, calcite, and sodium feldspar in the sandstone under acidic conditions, which causes the loss of internal cement and reduces the mechanical strength. Moreover, the cementitious reaction inside the sandstone becomes more violent as the pH value decreases, and the deterioration of the cementitious structure inside the rock becomes more obvious [14].

Figure 6 shows the relationship between the Young’s modulus of sandstone and pH values. As can be seen, the Young’s modulus of rock samples generally decreases linearly when the pH value decreases. After 10 drying–wetting cycles using pH = 6.0, 4.0, and 2.0 solutions, the Young’s modulus values of the sandstone specimens are 92.0%, 78.4%, and 66.7% at pH = 7.0.

The Poisson’s ratios of the specimens after the drying–wetting cycles at pH = 7.0, 6.0, 4.0, and 2.0 solutions are 0.19, 0.22, 0.20, and 0.21, respectively. With the enhanced acidity, the Poisson’s ratio of the sandstones generally shows an upward trend. Moreover, acidic corrosion promotes the softening of the internal structure of sandstones, thus improving their ductility.

**Table 2.** Mechanical parameters of sandstone after the dry–wet cycle of the acid solutions at different pH values

| pH  | Peak strength (MPa) | Peak strain (%) | Young’s modulus (GPa) | Poisson’s ratio |
|-----|---------------------|-----------------|------------------------|----------------|
| 7.0 | 48.7                | 0.45            | 12.5                   | 0.19           |
6.0  45.4  0.45  11.5  0.22
4.0  43.2  0.47  9.8   0.20
2.0  33.9  0.46  8.3   0.21

| pH | Peak strength / MPa | Young's modulus / GPa |
|----|---------------------|-----------------------|
| 7  | 12.5                | 9.5                   |
| 6  | 11.0                | 10.0                  |
| 5  | 10.5                | 11.0                  |
| 4  | 10.0                | 12.0                  |
| 3  | 9.5                 | 13.0                  |
| 2  | 9.0                 | 14.0                  |

**Figure 5.** Relationship between peak strengths and pH values of the sandstone specimens.

**Figure 6.** Relationship between the Young’s modulus and pH values of the sandstone specimens.

4. Characteristic stress

Many intensive studies have been conducted on the progressive failure of brittle rocks in laboratory compression tests [12]. Several stress thresholds have been proposed based on the progressive failure process of the rock, in order to characterize the strength of the rock, including crack closure stress (CCS), crack damage stress (CD), crack initiation stress (CI), and peak stress (UCS). As shown in Figure 7, in the initial stage of the stress–strain curve, the curve appears concave, which is due to the large porosity of sandstone. The pores and cracks inside the specimen are compacted in the initial stage of axial loading. Stress reaches CCS when the stress–strain curve turns into the linear elastic stage. Strength parameters, such as the Young’s modulus and Poisson’s ratio, of the sandstone specimens can be obtained from the stress–strain relationship at this stage. CI represents the axial stress corresponding to the end of the linear elastic stage, and the stress–strain curve shows a downward concave characteristic. As the axial stress continues to increase, the stress–strain curve begins to enter the stage of crack unsteady growth, in which CD appears. The internal cracks in the sandstone sample continue to expand under the loading of axial stress until they eventually penetrate to form micro-cracks. At this time, the axial stress reaches the maximum value, which is UCS. With regard to the process of determining the characteristic stress in the progressive failure process of rocks, researchers have conducted various explorations and proposed many methods to determine the characteristic stress. The methods for determining the characteristic stress in the current paper are as follows: determining CD using the volume strain method, determining CCS via the axial strain response method (ASR), and determining CI based on the lateral strain response method (LSR) [15], [19], [20].

4.1. Crack damage stress (CD)

According to the volume strain method proposed by Martin, there exists a maximum point on the curve of the volumetric strain–axial strain [15]. Before reaching the maximum point, the volume of the rock sample shows shrinkage with the axial stress loading; however, when exceeding the maximum point, the volume of the rock sample appears to expand. The axial stress corresponding to the maximum axial strain is the CD of the rock specimen. Figure 8 presents the volume strain–axial stress curves of specimens with different solutions of drying–wetting cycles based on the volume strain method.
Figure 7. Schematic diagram of the stress–strain curves for the progressive failure of rock specimens. (a) The different stages of crack closure and expansion, the change of crack volumetric strain, and volumetric strain response, are illustrated; (b) The schematic diagram showing different crack behaviors.

Figure 8. Relationship between volumetric strain and axial stress of the sandstone specimens.

Figure 9. Relationship between the crack closure stress (CCS) and pH value of the sandstone specimens.

According to the volumetric strain method, the corresponding CD values of the sandstone specimens with drying–wetting cycles for 10 times using different solutions are 33.1, 29.0, 28.3, and 20.6 MPa. As shown in Figure 9, as the acidic action increases, the CD of sandstone shows a decreasing trend. Thus, the acidic environment reduces the strength of cementite in sandstone, thus loosening the mineral particles.

4.2. Crack closure stress (CCS)
Peng [20] determined the CCS of the sandstone specimens according to ASR. After determining the damage stress of rocks using the volume strain method, we connected the damage stress point and origin on the stress–strain curve with a straight line. Then, we identified the point of the maximum axial strain difference between the stress–strain curve and the straight line. The axial stress corresponding to this point is the CD of each sandstone specimen.

Figure 10. Relationship between crack closure stress (CCS) and pH values of the sandstone specimens.

According to the experimental data, the axial stress–strain curves of the specimens that underwent drying–wetting cycles with different solutions are plotted in Figure 4. Meanwhile, the CCS of the specimens calculated according to the LSR method is shown in Figure 10. The CCS values of the specimens under the conditions of pH 7.0, 6.0, 4.0 and 2.0 solutions and drying–wetting cycles are 9.8, 7.3, 6.4, and 4.6 MPa, respectively. Overall, the CCS values of the specimens decrease with increasing acidity, as indicated by the fact that the CCS of the sandstone samples treated with the acidic solution at pH = 2.0 was reduced by 53.1% compared to the pH of 7.0. One possibility for this result is that the micro-cracks inside the sandstone sample spread throughout and formed large cracks. The chemical reaction of the cemented material around the micro-cracks also caused the bond strength to decrease, thus significantly attenuating the crack closure stress of the sandstone samples.

4.3. Crack initiation stress (CI)
The CI of the sandstone specimen is determined according to the lateral strain response method (LSR) proposed by Nicksiar and Martin [19]. Here, we connect the damage stress point and origin on the axial stress–lateral strain curve with a straight line. Then, we find the point where the maximum lateral strain difference exists between the axial stress–lateral strain curve and the straight line. The axial stress corresponding to this point is the CI of the sandstone specimen.
**Figure 11.** Axial stress–lateral strain curves of the sandstone specimens.

**Figure 12.** Relationship between the crack initiation stress (CI) and pH values of the sandstone specimens.

**Figure 13.** Curves of the lateral strain difference–axial stress of the sandstone specimens.

(a) pH = 7.0, (b) pH = 6.0, (c) pH = 4.0, (d) pH = 2.0.

Figure 11 shows the axial stress–lateral strain curve, and Figure 13 presents the lateral strain difference–axial stress curves of the sandstone specimens with different solution treatments. According to the LSR method, the CI values of the sandstone specimens with drying–wetting cycles of pH 7.0, 6.0, 4.0, and 2.0 solutions are 16.2, 14.5, 13.9, and 10.5 MPa, respectively. The CI of sandstone is about 31%–33% of UCS, which is consistent with the results reported by many scholars. Furthermore, as shown in Figure 12, the CI of the specimen continuously decreases with the increase of the acidity of the solution.

4.4. **Normalized characteristic stress**

Based on the above analysis, the CD, CI, CCS, and UCS of the sandstone specimens subjected to drying–wetting cycle treatment for 10 times using different solutions are obtained. The results are shown in Table 3. Moreover, the normalized characteristic stress values (characteristic stress/peak stress) of the samples can be calculated according to the characteristic stress values.

**Table 3.** Characteristic stress values of sandstone specimens subjected to different acidic drying–wetting cycles

| pH  | CCS (MPa) | CI (MPa) | CD (MPa) | UCS (MPa) |
|-----|-----------|----------|----------|-----------|
| 7.0 | 48.7      | 0.45     | 12.5     | 0.19      |
| 6.0 | 45.4      | 0.45     | 11.5     | 0.22      |
As shown in Figure 14, the normalized damage stress, normalized crack closure stress, and normalized crack initiation stress of the sandstone specimens generally show a decreasing trend with the increase of the acidic action. With the change from pH = 7.0 to pH = 6.0, the normalized damage stress and normalized crack closure stress decrease slightly. Moreover, the properties of the sandstone deteriorate due to the internal structure of the sandstone sample changed by the acidic action.

Figure 14. Relation between normalized characteristic stress and pH values.

5. Conclusion
(1) Under the condition of acidic drying–wetting cycle, the physical properties (e.g., P-wave velocity, peak intensity, and Young’s modulus of sandstone) are significantly reduced with the increase of acidic action.
(2) The characteristic stress values of the sandstone specimens with different solutions during the drying–wetting cycles were calculated based on the volumetric strain method, i.e., the ASR and LSR theoretical methods. The change law of characteristic stress (CCS, CI, and CD) of the sandstone samples under the action of acidic dry–wet cycle was also analyzed. Results revealed that the characteristic stress of sandstone continues to decrease with the increase of acidity and that the decrease from neutral solution to weakly acidic solution is most obvious. However, the normalized characteristic stress changes little with the effect of the acidic drying–wetting cycle.

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