

## Article

# Physical and Mechanical Properties Evolution of Coal Subjected to Salty Solution and a Damage Constitutive Model under Uniaxial Compression

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**Citation:** Wang, M.; Guo, Q.; Tian, Y.; Dai, B. Physical and Mechanical Properties Evolution of Coal Subjected to Salty Solution and a Damage Constitutive Model under Uniaxial Compression. *Mathematics* **2021**, *9*, 3264. <https://doi.org/10.3390/math9243264>

Academic Editor: Tinh Quoc Bui

Received: 5 November 2021

Accepted: 8 December 2021

Published: 16 December 2021

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**Abstract:** Many underground reservoirs for storing water have been constructed in China's western coal mines to protect water resources. Coal pillars which work as dams are subjected to a long-term soaking environment of concentrated salty water. Deterioration of the coal dam under the attack of the salty solution poses challenges for the long-term stability and serviceability of underground reservoirs. The evolution of the physical and mechanical properties of coal subjected to salty solutions are investigated in this paper. Coal from a western China mine is made to standard cylinder samples. The salty solution is prepared according to chemical tests of water in the mine. The coal samples soaked in the salty solution for different periods are tested by scanning electron microscope, nuclear magnetic resonance, and ultrasonic detector techniques. Further, uniaxial compression tests are carried out on the coal specimens. The evolutions of porosity, mass, microstructures of coal, solution pH values, and stress–strain curves are obtained for different soaking times. Moreover, a damage constitutive model for the coal samples is developed by introducing a chemical-stress coupling damage variable. The result shows that the corrosion effect of salty solution on coal samples becomes stronger with increasing immersion time. The degree of deterioration of the longitudinal wave velocity ( $v_p$ ) is positively correlated with the immersion time. With the increase in soaking times, the porosity of coal gradually increases. The relative mass firstly displays an increasing trend and then decreases with time. The peak strength and elastic modulus of coal decreases exponentially with soaking times. The developed damage constitutive model can well describe the stress–strain behavior of coal subjected to salty solution under the uniaxial compression.

**Keywords:** coal; deterioration characteristics; chemical-stress coupling factor; damage constitutive model

## 1. Introduction

Coal mining is normally accompanied by discharge of a large amount of water. This drained mine water with a high level of salt, in some cases, may damage ecosystem balance. Some theories and techniques, including the adsorbent method, evaporation, bioremediation, and irrigation of croplands, have been proposed and applied to treat the sodic–salty associated water from coal seams [1]. The coal resources of China are mainly distributed in western mining areas where the ecological environment is fragile and water resources are scarce. Large-scale coal mining could result in environmental problems, such as surface water waste and environmental pollution [2,3]. To protect and utilize water resources, some underground mines [4] built underground reservoirs in the underground

coal mines for water storage. However, mine water in some of these mines [4] has high salinity level with  $K^+$ ,  $Na^+$ ,  $Cl^-$ , and  $SO_4^{2-}$ , etc. As the dams of underground reservoirs, coal pillars are in a long-term soaking environment of concentrated saltwater that could significantly change the microstructure of coal and affect the performance of coal dams. The immersion of salty solutions poses a serious threat to the stability of the coal dams. Therefore, it is crucial to understand the mechanical behavior of coal subjected to saltwater for the design and management of underground coal dams.

In past decades, considerable research has been carried out on the water–rock interaction. The chemical solutions mainly affect the rocks through hydro-physical and hydro-chemical interactions. The physical effects of water on rocks include water lubrication, water wedge, etc. The presence of water promotes the dissolution of soluble salts, accelerates the hydrolysis of colloids, weakens the connection force between mineral particles, and finally reduces the strength of the rock. Scholars have conducted a lot of research on the influence of water on the physical and mechanical properties of rock materials. Dyke and Dobereiner [5] studied the influence of water content on the strength and deformation characteristics of sandstone and found that small changes in water content can significantly affect the mechanical response of the rock. The increase in water content promotes the propagation of microcracks and the occurrence of dilatancy, which results in a decrease in rock strength. Hawkins and McConnell [6] discussed the sensitivity of different types of sandstone to water. It showed that the water sensitivity of sandstone increases with the increase in the proportion of quartz and clay minerals, and the strength of sandstone is in an exponential relationship with water content. Vásárhelyi [7] carried out experiments on the physical and mechanical properties of dry and saturated limestone and found that the porosity, elastic modulus, uniaxial compressive strength, and tensile strength of limestone under saturated conditions are all 66% of the dry specimens. Erguler [8] conducted uniaxial compression tests and Brazilian splitting tests on clay-bearing rocks with different water content. With the increase in water content, the uniaxial compressive strength, elastic modulus, and tensile strength of the rock were reduced by 90%, 93%, and 90%, respectively, compared with the specimen in the dry state. Zhao [9] studied coal's dynamic tensile failure characteristics in dry and saturated states, and found that saturated coal samples have higher tensile strength compared with dry coal samples. Gu [10,11] tested the static and dynamic mechanical properties of coal samples under different water-bearing conditions. The results showed that with the increase in water content, the static mechanical parameters of coal samples deteriorated to a certain degree, while the dynamic mechanical parameters increased first and then decreased.

The chemical effects of water on rocks include ion exchange, dissolution, and hydrolysis. The water and rock chemical action changes the mineral composition in the rock mass and increases the pore structure, which leads to the change in macro-mechanical properties. M. G. Karfakis and M. Akram [12] discussed the influence of water–rock interactions on rock fracture toughness. Fench et al. [13] carried out triaxial compression tests on fractured quartz sandstones with different pH levels of NaCl and  $CaCl_2$ , and studied the effects of different solutions on the friction strength of sandstone fracture surfaces. Feng and Chen et al. [14–16] carried out experiments on the mechanical properties of sandstone under different corrosion conditions, discussed the microscopic failure mechanism of the rock under the action of corrosion, and carried out a quantitative analysis of the damage evolution inside the rock. Han et al. [17] conducted mechanical tests on sandstone after soaking in different chemical solutions and analyzed the corrosion effect of different solutions on the rock. Hutchinson and Johnson [18] studied the corrosion and degradation of limestone under the action of acid. Qiao [19] carried out uniaxial compression tests of sandstone after soaking in aqueous solutions with different ion concentrations, and analyzed the influence of water chemistry on the microstructure and macro-mechanical properties of the rock. Lin [20] studied the evolution of mechanical damage of rocks under the coupled chemical-stress conditions, and found that chemical corrosion increased the porosity of the rocks, which led to the decline of mechanical properties. Xie [21] tested the influence of

chemical degradation on the mechanical behavior of limestone and found that chemical degradation enhanced the deformation of rock and significantly increased the permeability. Li [22] employed the nuclear magnetic imaging technology and mechanical tests method to study the effect of chemical solutions on the degradation of the microstructure and mechanical properties of limestone.

However, most existing research on the effects of water and chemistry on rock mechanical behaviors were focused on rock, e.g., limestone, granite, sandstone, etc. Some studies on the influence of salty solution on coal have been carried out in surfactant absorption and mineral flotation. Ozdemir et al. [23] used a series of experimental measurement methods to study the influence of hypersaline on surface chemistry characteristics of coal flotation. They found that the hypersaline reduced the surface tension between coal particles and air bubbles. Zhang et al. [24] studied the effect of different ions on interfacial tension between water and kerosene and found that the surface tension decreased with the increasing salt concentration. Ni et al. [25] studied the effect of NaCl-SDS compound solution on the wetting performance of coal. It found that the addition of sodium salt can efficiently decrease the surface tension of coal and improve the wettability. To date, research on coal's physical and mechanical properties subjected to salty solutions is rarely reported. The physics and mechanisms behind coal damage affected by salty mine water are unclear, bringing difficulties to design, management, and stability control of coal dams in underground reservoirs.

This paper aims to study the evolution of the physical and mechanical properties of coal subjected to salty solution. Firstly, coal samples from Ningdong mining area in western China are prepared to standard cylinders. A salty solution is prepared based on chemical test results of mine water. The scanning electron microscope and nuclear magnetic resonance technology are employed to study the changes in porosity, mass, and microstructure of coal samples for different soaking times. Then, uniaxial compression tests are carried out to obtain the stress–strain curves of coal samples for different soaking times. The effects of salty solution on the damage of coal samples are discussed. Moreover, a damage constitutive model for the coal samples is developed by introducing a chemical–stress coupling damage variable. The developed model is verified with the stress–strain curves from uniaxial compression tests.

## 2. Experimental Materials and Methods

### 2.1. Preparation of Coal Samples

The coal used in the test is taken from Lingxin coal mine in Ningdong Mining Area, China. The coal samples are drilled from the same coal block to ensure uniformity. According to the ISRM rock preparation standard [26], the coal samples are made into a standard cylinder with a diameter of 50 mm and a height of 100 mm. The unevenness of the two end faces should not exceed  $\pm 0.05$  mm. The end face is perpendicular to the axis of the rock sample, and the allowable deviation is  $\pm 0.25^\circ$ .

### 2.2. Salt Solution

Groundwater is a complex chemical solution containing various ionic components. The cations in the mine water retrieved from the Lingxin mining area were determined by using inductively coupled plasma atomic emission spectroscopy (ICP-OES 730, Agilent, Santa Clara, CA, USA) and the anions were tested by using Chromatograph (LC-2010 PLUS, SHIMADZU, Kyoto, Japan). The chemical test results are presented in Table 1. The main ion components of the mine water are  $\text{Na}^+$ ,  $\text{SO}_4^{2-}$ , and  $\text{Cl}^-$  and the calculated concentration ratio is about 5:1:3. In this study, the mixed anions of  $\text{SO}_4^{2-}$ - $\text{Cl}^-$  and the cation  $\text{Na}^+$  are selected to study the degradation effect of the salty solution on coal samples. Since the water–coal reaction is a long-term process, we used a higher ion concentration in the configuration solution than that in mine water to shorten the experimental period. The ratio of the cation ( $\text{Na}^+$ ) to mixed anions ( $\text{SO}_4^{2-}$ - $\text{Cl}^-$ ) is the same as the ratio in mine water. In the final salty solution, the concentration of sulfate ions is 0.1 mol/L, and the

solution pH is 9. The chemical reagents used in this study are sodium chloride ( $\geq 99.5\%$  purity), sodium sulfate ( $\geq 99\%$  purity), and sodium hydroxide ( $\geq 96\%$  purity) that are all made by Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). High-purity distilled water is used throughout the experiment.

**Table 1.** Chemical test results of mine water.

| PH   | TDS  | K <sup>+</sup> | Na <sup>+</sup> | Ca <sup>2+</sup> | Cl <sup>−</sup> | SO <sub>4</sub> <sup>2−</sup> |
|------|------|----------------|-----------------|------------------|-----------------|-------------------------------|
|      | mg/L | mg/L           | mg/L            | mg/L             | mg/L            | mg/L                          |
| 8.79 | 4720 | 10.7           | 1350            | 86               | 1230            | 1145                          |

### 2.3. Experimental Procedure

After the coal samples were prepared, the longitudinal wave velocities of coal samples were measured to ensure uniformity of samples. The coal samples with close wave velocities were divided into seven groups (US1-US6 and P) according to the immersion time. Each group contained three samples, and a total of 21 samples were used for further experiments. The sample group US1-US6 were used for mechanical tests and the sample group P was used for physical tests. The coal samples were dried before immersion and the original mass were weighed. The solution was regularly agitated during the soaking process to make the coal and solution fully interact. During the soaking process, the pH value of the solution, the mass, the longitudinal wave velocity, and the porosity of the coal samples were measured, and the experimental temperature was 20 °C. The measurement time interval was determined according to the change rate of pH value of the salty solution. When the pH value of the solution becomes stable, it is considered that the water–coal interaction has reached a steady state. An acidity meter PHS-3E from China Shanghai INESA Scientific Instrument Co. Ltd. was used to measure the pH value of the solution. The accuracy of the acidity meter is 0.01. The longitudinal wave velocity was tested by a ZBL-U5200 non-metallic ultrasonic detector produced by China Beijing ZBL SCI & TECH Co. Ltd. An electronic scale with accuracy of 0.01 g was used for the measurement of coal mass. The sample group P was chosen for the mass and the longitudinal wave velocity test throughout the whole immersion time.

The coal sample P-2 was selected for the porosity test, and the evolution law of porosity throughout the immersion period was analyzed. The low-field nuclear magnetic resonance analyzer produced by China Suzhou Newmarket analytical instrument company was applied for porosity tests.

In order to understand the microscopic mechanism of the effect of salty solution on coal's pore feature, SEM technology was used to analyze the microstructure of coal. The coal was cut into a 1 cm × 1 cm × 1 cm square piece to meet the requirements of the SEM sample size. In this study, the SEM of ZEISS EVO 18 (Jena, Germany) was used for the microscopy experiment. The related parameters are: acceleration voltage, 200 V–30 Kv; magnification times, 5–10<sup>6</sup> times; focused working distance, 2–145 mm; and sample size, diameter ≤ 250 mm, height ≤ 145 mm.

For the uniaxial compression tests, the MTS815 rock mechanics testing machine was applied. The maximum axial load of the MTS815 testing machine is 2700 kN. The ranges of the axial and circular extensometers are respectively 5 mm and 8 mm. A combination of stress loading and hoop displacement loading was adopted in the test. The loading rate is 100 N/s. When the axial load increases to 30 KN, it converts to displacement loading with a 0.01 mm/min loading rate. Figure 1 shows the detailed flow of experiments.

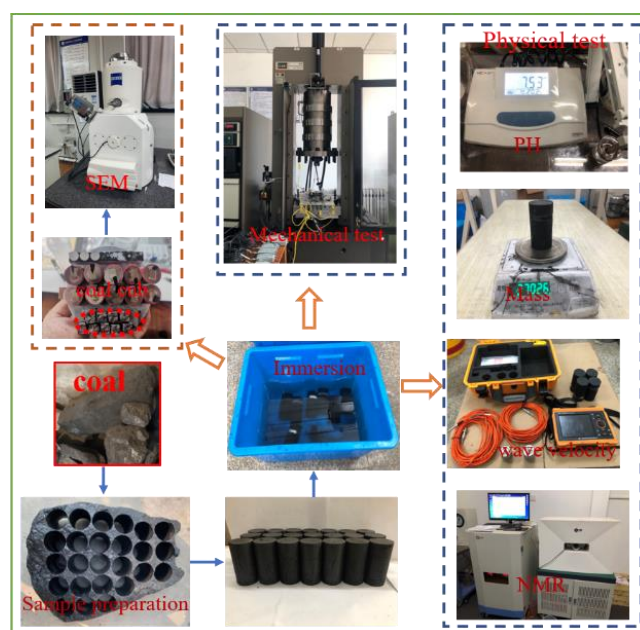


Figure 1. Diagrammatic sketch showing the flow of experiments.

### 3. Results and Discussion

#### 3.1. Changes in PH Values of Salt Solution

Figure 2 shows the relationship between the soaking times and pH value of the solution. It can be seen that as the soaking time increasing, the pH value of the solution gradually tends to be neutral, indicating that the PH value of the solution has the ability of self-balancing during the process of the water–rock interaction. The pH value of the solution first changed very obviously, and then the changing rate gradually decreased. This indicates that the water–rock interaction is time dependent, i.e., the water–rock reaction will gradually weaken with the increase in the soaking time, and eventually tend to be stable.

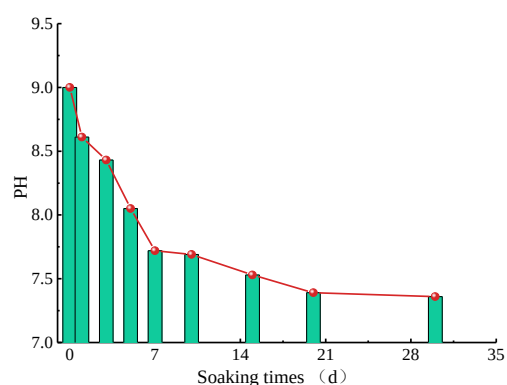


Figure 2. The relationship curve between soaking times and PH values of solution.

#### 3.2. Variation Law of Longitudinal Wave Velocity of Coal Samples

Many studies have shown that the longitudinal wave velocity of rock samples is sensitive to the development of microstructures such as internal pores and defects [27–32]. In order to eliminate the errors caused by the differences in samples, the longitudinal wave velocity of the same sample was tested during the immersion period. Therefore, the changes in longitudinal wave velocity can be used to characterize the influence of salty solution on the damage to the internal microstructure of the coal samples.

The test results indicated that the longitudinal wave velocity of coal samples displays different degrees of deterioration with the increasing soaking times. In this paper, the change rate of longitudinal wave velocity ( $v_{cr}$ ) was used to characterize the damage law

of  $v_p$  caused by chemical corrosion. The larger the value of  $v_{cr}$ , the higher the degree of damage. The expression of the change rate of  $v_{cr}$  is shown as follows:

$$v_{cr} = \frac{|v_{pt} - v_{p0}|}{v_{p0}} \times 100\% \quad (1)$$

In the above,  $v_{p0}$  and  $v_{pt}$  are the longitudinal wave velocities of coal samples before and after the different soaking times.

Figure 3 displays the relationship between soaking times and  $v_{cr}$  of sample P-2. As the soaking time increases, the longitudinal wave velocities of the coal sample exhibit a deteriorating trend. At the beginning of the test, the longitudinal wave velocity of coal samples deteriorated at a high rate and the water–coal reaction was violent, which caused the increase in pores of the coal sample. Macroscopically, the longitudinal wave velocity decreased to varying degrees. Table 2 displays the mass and  $v_p$  of coal samples under different soaking times.

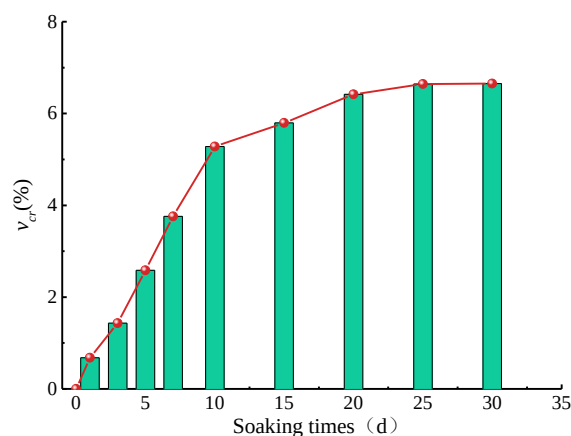


**Figure 3.** The relationship curve between the soaking times and the change rate of longitudinal wave velocities.

**Table 2.** The mass and longitudinal wave velocity ( $v_p$ ) of coal samples under different soaking times.

| Soaking Times/d | P-1    |                                   | P-2    |                                   | P-3    |                                   |
|-----------------|--------|-----------------------------------|--------|-----------------------------------|--------|-----------------------------------|
|                 | Mass/g | $v_p/\text{km}\cdot\text{s}^{-1}$ | Mass/g | $v_p/\text{km}\cdot\text{s}^{-1}$ | Mass/g | $v_p/\text{km}\cdot\text{s}^{-1}$ |
| 0               | 257.79 | 1.83                              | 255.32 | 1.76                              | 253.07 | 1.80                              |
| 0.25            | 261.15 | -                                 | 258.85 | -                                 | 256.12 | -                                 |
| 0.5             | 265.33 | -                                 | 263.13 | -                                 | 261.18 | -                                 |
| 1               | 270.91 | 1.82                              | 268.24 | 1.75                              | 265.86 | 1.79                              |
| 3               | 269.92 | 1.80                              | 267.06 | 1.73                              | 264.99 | 1.77                              |
| 5               | 269.54 | 1.79                              | 267.30 | 1.71                              | 264.44 | 1.76                              |
| 7               | 269.04 | 1.76                              | 266.50 | 1.69                              | 264.49 | 1.73                              |
| 10              | 268.40 | 1.73                              | 265.71 | 1.67                              | 263.32 | 1.71                              |
| 15              | 268.02 | 1.73                              | 265.73 | 1.66                              | 262.73 | 1.70                              |
| 20              | 266.85 | 1.72                              | 264.47 | 1.65                              | 262.04 | 1.69                              |
| 25              | 266.80 | 1.71                              | 264.28 | 1.64                              | 262.00 | 1.68                              |
| 30              | 266.77 | 1.71                              | 264.26 | 1.64                              | 262.02 | 1.68                              |

### 3.3. Mass Changes of Coal Samples

During the immersion process, the water–coal reaction dissolves the mineral components in the coal samples and finally changes the mass of the coal sample. The mass differences between the original dried samples and saturated samples with different soaking times were analyzed, which can indirectly reflect the water–coal process’s degradation law. The prepared coal samples were dried for 24 h with temperature of 50 °C and the

original mass  $m_0$  were weighed. After different soaking times, the coal samples were taken out from the salt water, the moisture on the surface of the samples was wiped off, and the mass of the saturated samples ( $m_t$ ) were weighed. The relationship between the  $m_0$  and  $m_t$  is shown as follows:

$$\begin{cases} m_0 + m_l - m_r = m_t \\ \Delta m = m_t - m_0 = m_l - m_r \end{cases} \quad (2)$$

where  $m_l$  represents the mass of pore water inside the coal sample;  $m_r$  represents the mass of the dissolved substance.

Figure 4 shows the relationship between soaking times and the relative mass difference of sample P-2. It can be seen that the relative mass difference of the coal sample increases rapidly at the beginning of the test, then gradually decreases. At the initial state of immersion, the solution diffuses through the pores accompanied by the water–coal reaction, but the mass of the immersed solution was obviously greater than that of the dissolved substance, which led to a rapid increase in the relative mass difference. As the soaking time increased, the coal sample reached a saturated state. The pore volume of the coal sample gradually increased with the dissolution, and the immersed solution in the pores continued to increase. However, the strong water–coal reaction made the mass of dissolved substance greater than that of the added immersion solution, which decreased the relative mass difference. After that, the interaction between solution and coal sample gradually weakened, and the variation range of relative mass difference gradually reduced.

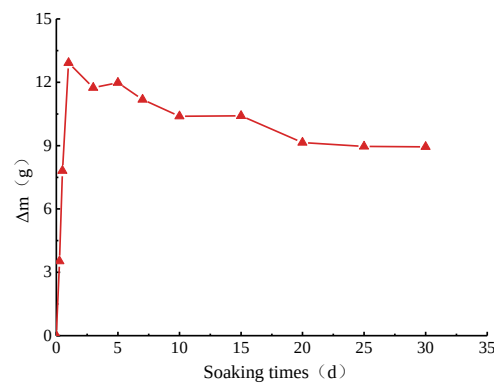


**Figure 4.** The relationship between  $\Delta m$  and soaking times.

### 3.4. Porosity of Coal Samples

The nuclear magnetic resonance (NMR) method was used to observe the change of porosity and pore size distribution of coal samples under different soaking times. The signal source in the NMR test is hydrogen ion. Based on the hydrogen ion signal detected in the pores, the analysis software obtains the relaxation time ( $T_2$ ) and the initial magnetic field vectors of pores with different sizes through a special sequence (CPMG). Finally, the distribution of the relaxation time can be obtained through inversion. Different relaxation times represent different sizes of pores and the larger relaxation times represent larger pore sizes. The area of the relaxation peak in the  $T_2$  curves reflects the number of pores [33]. Equation (3) represents the relationship between  $T_2$  and parameters of pores [33].

$$1/T_2 = \rho(S/V) = F_s(\rho/r) \quad (3)$$

where  $T_2$  represents the inversion relaxation time, ms;  $\rho$  represents lateral relaxation density,  $\mu\text{m}/\text{ms}$ ;  $S$  represents the surface area of pore,  $\text{cm}^2$ ;  $V$  represents pore volume,  $\text{cm}^3$ ;  $r$  represents pore size, nm; and  $F_s$  represents shape factor of pore.

Figure 5 exhibits the  $T_2$  distribution of coal samples under different soaking times. The pore structure of coal is divided into three regions: the adsorption pores with a pore size of 1–100 nm (corresponding  $T_2$  smaller than 2.5 ms), seepage pores with a pore size of 100–10,000 nm (corresponding  $T_2$  larger than 2.5 ms and smaller than 50 ms), and fractures

with a pore size larger than 10,000 nm (corresponding  $T_2$  larger than 50 ms) [34–37]. Figure 5 shows that the  $T_2$  distribution of coal under different soaking times has a bimodal characteristic. After the immersion in salty solution, the  $T_2$  distribution curves of coal move towards the right, indicating that the immersion effect increases the connectivity of the micropores and results in the appearance of pores with larger sizes. The area of  $T_2$  distribution curves corresponding to the relaxation times of 0–2.5 ms, 2.5–50 ms, and 50 ms–1000 ms were calculated and the values represent the number of pores. Table 3 displays the pore number of coal samples after different soaking times. It is obvious that the number of seepage pores and fractures increased with the increase in soaking times. While the number of adsorption pores exhibits a trend of rapid increase first and then a slow decline. It is mainly because the small pores gradually connect to form larger pores in the later stage of immersion.

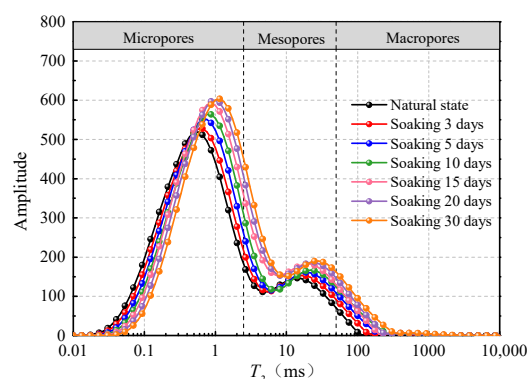


Figure 5. The  $T_2$  distribution curves of coal samples.

Table 3. Number of pores in coal samples after different soaking times.

| Soaking Times/d | Porosity/% | Adsorption Pores | Seepage Pores | Fractures |
|-----------------|------------|------------------|---------------|-----------|
| 0               | 21.28      | 9809             | 2570          | 213       |
| 3               | 21.91      | 9819             | 2765          | 354       |
| 5               | 22.96      | 10,041           | 2983          | 517       |
| 10              | 23.66      | 10,062           | 3172          | 688       |
| 15              | 24.88      | 10,385           | 3749          | 710       |
| 20              | 25.25      | 10,208           | 3982          | 807       |
| 30              | 25.58      | 9915             | 4210          | 1026      |

Under the effect of the water–coal interaction, the minerals in the coal sample dissolved, leading to increased porosity. Figure 6 shows the relationship curve between soaking times and porosity of coal sample.

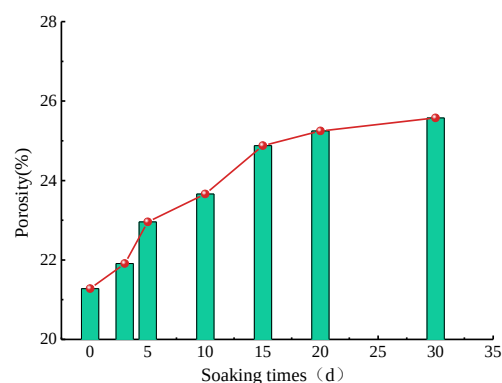


Figure 6. The change law of porosity as a function of soaking times.

### 3.5. Microscopic Morphological Characteristics of Coal Samples

The structure of coal is very complex due to the many mineral components in the coal. During the immersion process, the salty solution and the coal undergo a series of water–rock reactions, which lead to the dissolution of minerals, changes in the microscopic structure, and the increase in microcracks inside the samples.

It can be seen from Figure 7 that a large number of mineral particles are distributed on the surface of the coal sample under the natural state. At the same time, it can be observed that microfractures and micropores exist on the surface of coal. Most pores are blocked by mineral components, resulting in poor connectivity between pores. After being soaked for different times, the coal samples showed different degrees of corrosion, and the microscopic morphology changed to a certain extent. As the immersion time increases, the number of mineral particles on the surface of the coal samples decreases significantly. The mineral particles originally embedded in the coal body were corroded into holes of different sizes. Under the effect of the salty solution, microfractures gradually appeared and connected with the internal structure. The number of pores and fractures in the coal samples increased after being soaked with the salty solution. At the same time, the strength of the coal samples will gradually decrease with the increasing number of pores and cracks.

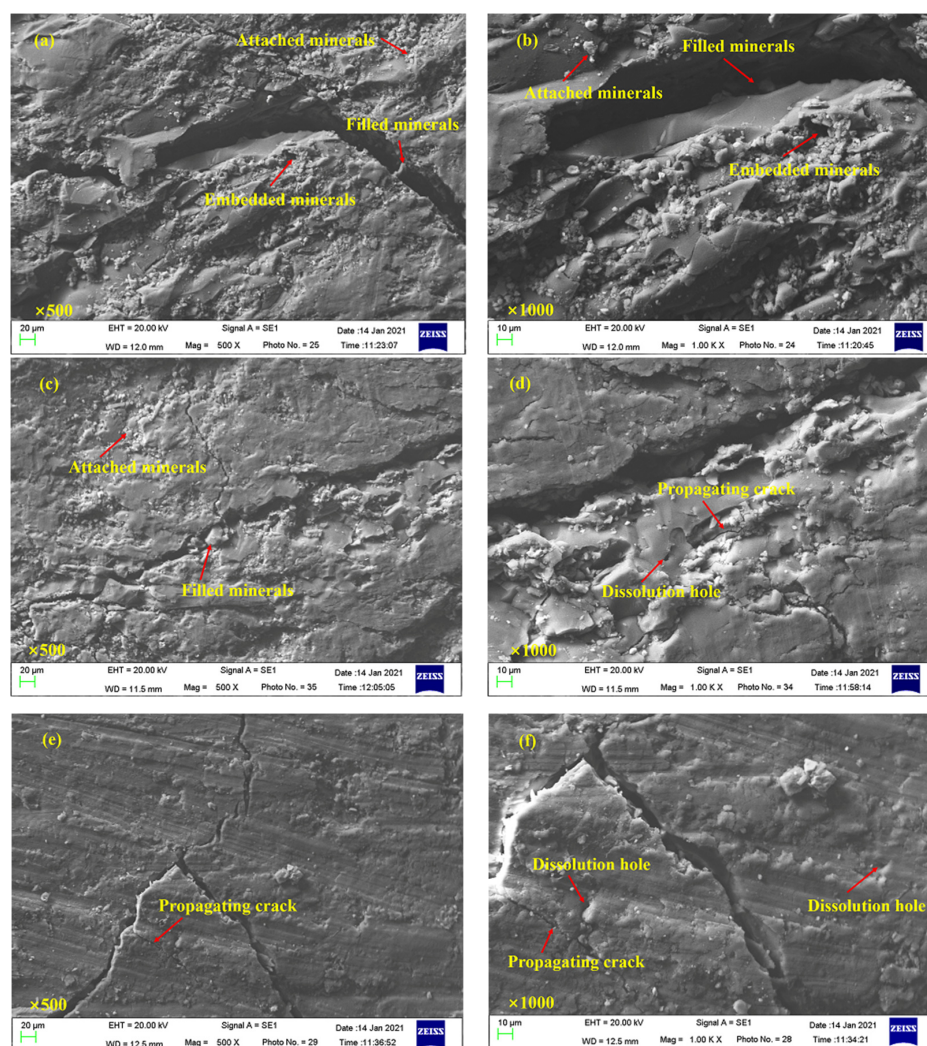


**Figure 7.** Microscopic morphology of coal samples after soaking for different times: (a,b) represent natural state; (c,d) soaking for 10 days; and (e,f) soaking for 30 days.

### 3.6. Mechanical Properties of Coal Samples

In the mechanical test, the stress and the strain of coal samples during the failure process were obtained. The stress–strain curves of coal samples for different soaking times (i.e., ST) are illustrated as Figure 8. The mechanical parameters calculated according to the stress–strain curve are shown in Table 4. It can be seen from Figure 8 that the compaction stage of the coal samples after immersion is longer than that of the natural state. As the soaking time increases, the compaction stage becomes longer. This is mainly because the internal defects such as pores and microfractures increased with the soaking time and more defects result in longer initial compaction stage of the stress–strain curve.

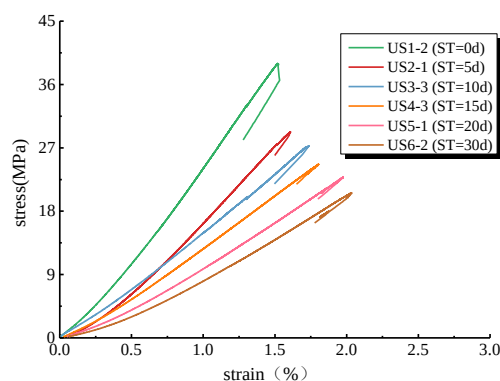


Figure 8. The stress–strain curve of coal samples.

Table 4. Mechanical parameters of coal samples.

| Sample No. | Soaking Time (d) | Peak Strength (MPa) | Elastic Modulus (GPa) | Failure Strain (%) |
|------------|------------------|---------------------|-----------------------|--------------------|
| US1-1      | 0                | 40.980              | 2.957                 | 1.483              |
| US1-2      | 0                | 38.974              | 2.865                 | 1.519              |
| US1-3      | 0                | 37.584              | 2.651                 | 1.538              |
| US2-1      | 5                | 29.269              | 2.172                 | 1.609              |
| US2-2      | 5                | 32.427              | 2.361                 | 1.581              |
| US2-3      | 5                | 27.985              | 1.886                 | 1.664              |
| US3-1      | 10               | 26.087              | 1.785                 | 1.763              |
| US3-2      | 10               | 27.953              | 1.603                 | 1.709              |
| US3-3      | 10               | 27.266              | 1.636                 | 1.737              |
| US4-1      | 15               | 23.758              | 1.508                 | 1.874              |
| US4-2      | 15               | 25.481              | 1.545                 | 1.792              |
| US4-3      | 15               | 24.663              | 1.475                 | 1.804              |
| US5-1      | 20               | 22.806              | 1.250                 | 1.976              |
| US5-2      | 20               | 23.469              | 1.347                 | 1.895              |
| US5-3      | 20               | 21.568              | 1.257                 | 1.983              |
| US6-1      | 30               | 21.357              | 1.166                 | 2.113              |
| US6-2      | 30               | 20.605              | 1.235                 | 2.034              |
| US6-3      | 30               | 18.624              | 1.013                 | 2.164              |

Figure 9 displays the relationship between soaking times and several mechanical parameters of coal samples. It appears that the peak strength and elastic modulus of the coal samples gradually decrease with the soaking time, while the failure strain exhibits an increasing trend with soaking time. The reduction in the peak strength of coal under the salty solution immersion is related to the adsorption of ions or molecules in the solution to the coal surface. The adsorbed ions or molecules on the coal surface reduce the surface energy and its fracture strength. The result manifests that the immersion of salty solution significantly influences the mechanical properties at the initial state, and the declining amplitude becomes smaller with the increasing soaking time.

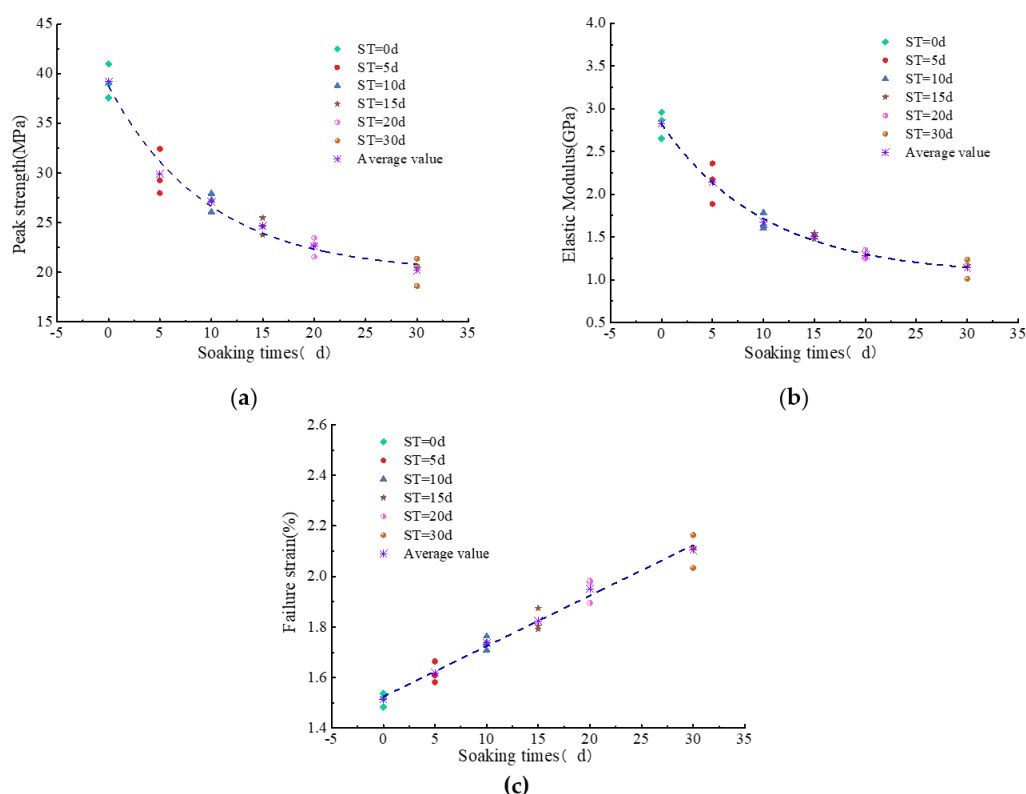


**Figure 9.** The relationship between soaking times and mechanical parameters of coal: (a) peak strength; (b) elastic modulus; and (c) failure strain.

#### 4. Statistical Damage Constitutive Relationship of Coal Samples under the Coupling Action of Salty Solution and Uniaxial Compression

##### 4.1. Damage Constitutive Relationship and Damage Evolution Equation under Uniaxial Compression

According to the principle of strain equivalence [38,39], the strain produced by the damaged material under the stress  $\sigma$  is equivalent to the strain produced by the undamaged material under the effective stress  $\sigma'$ .

$$\varepsilon = \frac{\sigma}{E'} = \frac{\sigma'}{E} = \frac{\sigma}{E_0(1 - D_s)} \quad (4)$$

In the above,  $\sigma$  and  $\sigma'$  are nominal stress and effective stress, respectively;  $\varepsilon$  is strain;  $E_0$  is the elastic modulus of the sample in the initial state;  $E'$  is the elastic modulus of the sample in a damaged state; and  $D_s$  is the damage variable under loading.

According to Equation (4), the damage constitutive model of coal samples during uniaxial compression can be obtained as follows:

$$\sigma = E\varepsilon(1 - D_s) \quad (5)$$

The rock material contains various defects, and the microstructure is nonuniform. The random distribution of these defects in the rock causes the damage to distribute randomly inside the rock during the compression process, resulting in great differences in the mechanical properties of the rock [40,41]. It can be considered that the mechanical properties of the rock are a random variable. The rock can be divided into micro-element bodies containing several defects, and the relationship between the statistical distribution density of material failure and the damage variable is assumed as in [41].

$$dD_s = P(\varepsilon)d\varepsilon \quad (6)$$

There,  $P(\varepsilon)$  is a measure of the damage rate of the micro-element body during the loading process and  $F$  is the strength parameters of rock micro-element body.

Assuming that the strength of the micro-element body obeys the Weibull distribution during the loading process, the probability density function can be expressed as in [41].

$$P(\varepsilon) = \frac{m}{\varepsilon_0} \left( \frac{\varepsilon}{\varepsilon_0} \right)^{m-1} \exp \left[ - \left( \frac{\varepsilon}{\varepsilon_0} \right)^m \right] \quad (7)$$

In the above,  $\varepsilon_0$  is the average value of the strength of the micro-element body, and  $m$  is the shape factor of the distribution function, representing the uniformity of the rock material.

Substituting Equation (7) into Equation (6), the internal damage evolution equation during compression can be obtained as:

$$dD_s = P(\varepsilon)d\varepsilon = \frac{m}{\varepsilon_0} \left( \frac{\varepsilon}{\varepsilon_0} \right)^{m-1} \exp \left[ - \left( \frac{\varepsilon}{\varepsilon_0} \right)^m \right] d\varepsilon \quad (8)$$

$$\begin{aligned} D_s &= \int_0^\varepsilon \frac{m}{\varepsilon_0} \left( \frac{\varepsilon}{\varepsilon_0} \right)^{m-1} \exp \left[ - \left( \frac{\varepsilon}{\varepsilon_0} \right)^m \right] d\varepsilon \\ &= 1 - \exp \left[ - \left( \frac{\varepsilon}{\varepsilon_0} \right)^m \right] \end{aligned} \quad (9)$$

Substituting Equation (9) into Equation (5), the damage constitutive relation of rock under uniaxial compression is:

$$\sigma = E\varepsilon(1 - D_s) = E\varepsilon \exp \left[ - \left( \frac{\varepsilon}{\varepsilon_0} \right)^m \right] \quad (10)$$

The stress–strain relationship curve of the rock should meet the following boundary conditions:

$$\begin{cases} \varepsilon = 0, \sigma = 0 \\ D_s = 0, d\sigma/d\varepsilon = 0 \\ \sigma = \sigma_p, \varepsilon = \varepsilon_p \\ \varepsilon = \varepsilon_p, d\sigma/d\varepsilon = 0 \end{cases} \quad (11)$$

In the above,  $\sigma_p$  and  $\varepsilon_p$  are the peak stress and peak strain of the rock, respectively.

By combining Equations (10) and (11), the following Equation can be obtained:

$$\begin{cases} m = \frac{1}{\ln \left( \frac{E\varepsilon_p}{\sigma_p} \right)} \\ \varepsilon_0 = \frac{\varepsilon_p}{(1/m)^{1/m}} \end{cases} \quad (12)$$

Substituting Equation (12) into Equation (9), the damage evolution Equation of the rock is obtained as:

$$D_s = 1 - \exp \left[ - \frac{1}{m} \left( \frac{\varepsilon}{\varepsilon_p} \right)^m \right] \quad (13)$$

Substituting Equation (13) into Equation (10), the damage constitutive Equation of the rock can be obtained:

$$\sigma = E\varepsilon(1 - D_s) = E\varepsilon \exp \left[ - \frac{1}{m} \left( \frac{\varepsilon}{\varepsilon_p} \right)^m \right] \quad (14)$$

#### 4.2. The Chemical Damage Variable under the Action of Salty Solution

The damage of the coal sample after immersion in salt solution includes not only the load damage, but also the chemical damage caused by the immersion. According to the

principle of strain equivalence, the constitutive relationship of the internal damage of the coal sample after immersion in the salt solution is expressed as:

$$\sigma = (1 - D_c)E_0\varepsilon \quad (15)$$

There,  $E_0$  is the elastic modulus of a sample in the initial state and  $D_c$  is defined as the chemical damage variable under salt solution immersion.

According to Equation (15), the expression of  $D_c$  can be obtained:

$$E_c = (1 - D_c)E_0 \quad (16)$$

$$D_c = 1 - \frac{E_c}{E_0} \quad (17)$$

In the above,  $E_c$  is the elastic modulus of a coal sample after immersion in salty solution.

Figure 10 shows the relationship between the average values of chemical damage variable and immersion time. With the increase in the immersion time, the chemical damage variable of the coal sample firstly grows rapidly, and then the growth rate gradually slows down. By fitting the test data of the damage variable, the relationship expression between the chemical damage variable and the immersion time is obtained as shown in Equation (18). Under the action of the salty solution, the chemical damage variable of the coal sample is in an exponential relationship with the immersion time and the correlation coefficient is 0.977. The increase in immersion time causes more accumulated damage inside the coal sample, resulting in the deterioration of the coal sample's compressive strength.

$$D_c = 0.582 - 0.712 \times 0.862^t \quad R^2 = 0.977 \quad (18)$$

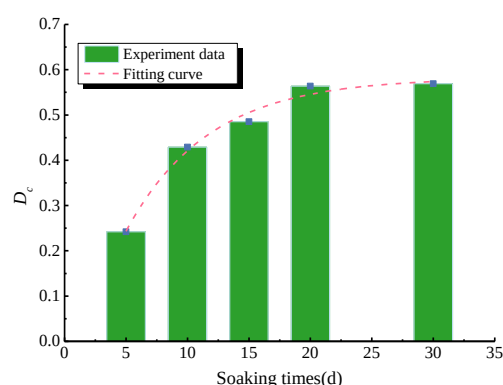


**Figure 10.** The relationship curve between average value of chemical damage variable and soaking times.

#### 4.3. The Damage Evolution Law under the Coupling Action of Uniaxial Compression and Salty Solution Immersion

The failure of the salty solution immersed coal sample under the action of the uniaxial load is caused by the mutual coupling and mutual influence of chemical damage and load damage. For the coal sample after soaking in salty solution, the total damage consists of two parts. The chemical damage caused by the salty solution immersion is considered as the first type of damage, and the damage caused by the load is regarded as the second type of damage.

$$\sigma = (1 - D_s)E_c\varepsilon \quad (19)$$

Substituting Equation (16) into Equation (19), the damage constitutive relationship of the coal sample under the coupling effect of salty solution immersion and uniaxial load can be obtained.

$$\sigma = (1 - D_s)(1 - D_c)E_0\varepsilon \quad (20)$$

The total damage variable  $D$  under the coupling action of chemical and load is expressed as Equation (21):

$$D = D_s + D_c - D_s D_c \quad (21)$$

Combining Equations (13), (17), and (21), the final form of total damage variable  $D$  is shown as Equation (22):

$$D = 1 - \frac{E_c}{E_0} \exp \left[ -\frac{1}{m} \left( \frac{\varepsilon}{\varepsilon_p} \right)^m \right] \quad (22)$$

The typical damage evolution curve under the coupling action of salty solution corrosion and uniaxial compression is shown in Figure 11. The damage degree of the specimen gradually increases with the increase in axial strain. The damage evolution of the coal sample under the coupling action of salty solution corrosion and load has obvious nonlinear characteristics. In the initial stage of compression, the internal microcracks and pores of the specimen are compressed. The degree of damage in the compaction stage is relatively small, resulting in a linear damage evolution curve. The continued action of the external load leads to the expansion of the microcracks, and increases the damage of the internal structure. Finally, crack penetration caused the specimen to be destroyed. Comparing the length of the linear section of the damage evolution curve, it is found that the length of the linear section of the curve gradually increases with the increase in the immersion time. It indicates that the porosity inside the coal sample increases with the increase in immersion time, resulting in the linear segment being more pronounced.

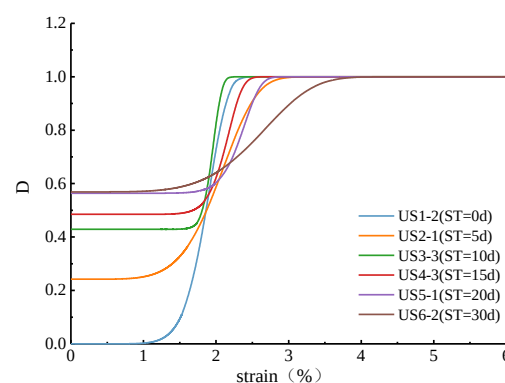


**Figure 11.** The typical damage evolution curve.

#### 4.4. Damage Constitutive Model Considering Chemical-Stress Coupling Factor

Combining Equations (12) and (20)–(22), the damage constitutive equation of the coal sample under the coupling action of chemical and uniaxial load can be expressed as Equation (23):

$$\begin{cases} \sigma = E_c \varepsilon \exp \left[ -\frac{1}{m} \left( \frac{\varepsilon}{\varepsilon_p} \right)^m \right] \\ m = \frac{1}{\ln \frac{E_c \varepsilon_p}{\sigma_p}} \\ \varepsilon_0 = \varepsilon_p m^{1/m} \end{cases} \quad (23)$$

In order to verify the damage constitutive model of the coal sample under the coupling effect of salty solution immersion and the uniaxial load, the stress–strain curve of the uniaxial test was compared with the curve obtained by the damage constitutive which is expressed as Equation (23). The damage constitutive curve is shown as the dotted line in Figure 12. It is found that the unmodified damage constitutive relationship curve is in poor agreement with the test curve. The characteristics of the stress–strain curve caused by the corrosion of the salty solution are not well described by using the unmodified damage constitutive (Equation (23)). Further, a chemical–stress coupling factor  $\mu$  was proposed to modify the damage constitutive. Literature [42] has introduced a thermal–mechanical coupling factor in the damage constitutive model to display the damage process of rock

under high temperatures. Like the rock treated with high temperature, the number of pores and cracks in the coal sample increased under the effect of the corrosion of salty solution. This leads to an increase in the length of the compaction stage. In this study, the expression of chemical-stress coupling factor  $\mu$  refers to that in literature [42]. The expression of coupling factor  $\mu$  and modified damage constitutive model are expressed as follows:

$$\mu = \mu_0 + A \exp \left[ -2(\varepsilon - \varepsilon_p)^2 / \omega^2 \right] / \omega \sqrt{\pi/2} \quad (24)$$

$$\sigma = \mu(1 - D_s)(1 - D_c)E_0\varepsilon \quad (25)$$

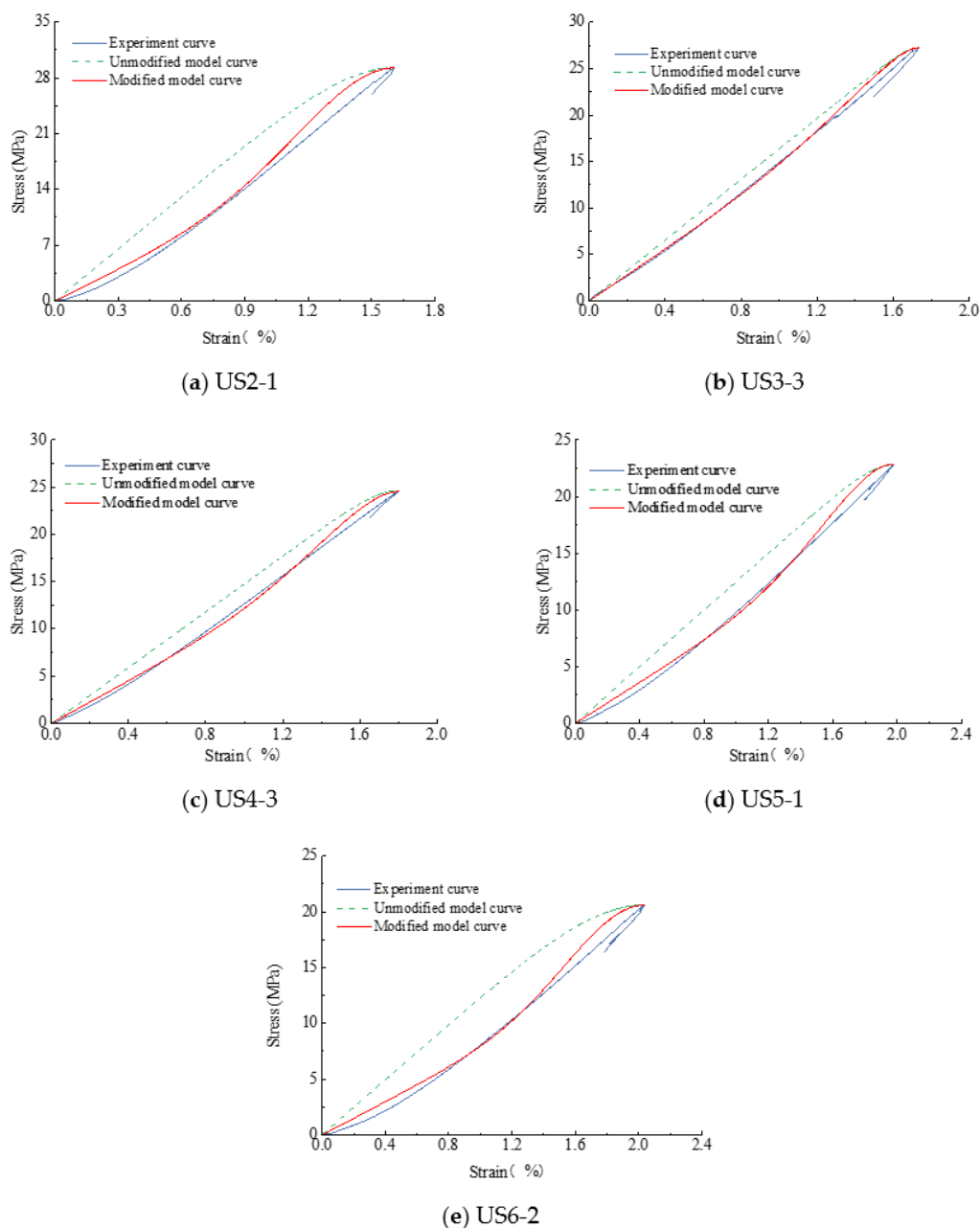


**Figure 12.** Comparison of stress–strain test curve and theoretical curve: (a) ST = 5 d, (b) ST = 10 d, (c) ST = 15 d, (d) ST = 20 d, and (e) ST = 30 d.

In the above,  $\varepsilon_p$  is the peak strain,  $\mu_0$  is the lower limit of the chemical-stress coupling factor,  $A$  is the integrated area of the function curve above the baseline, and  $\omega$  is the standard deviation of the function, which indicate the concentration of the chemical-stress

coupling effect. The related parameters ( $\mu_0$ ,  $A$ ,  $w$ ) in coupling factor  $\mu$  need to be determined by fitting the experimental data. The fitting parameters of coupling factor  $\mu$  are shown in Table 5.

**Table 5.** Fitting parameters in coupling factor  $\mu$ .

| Sample | $\varepsilon_c/10^{-2}$ | $A/10^{-2}$ | $w/10^{-2}$ | $\mu_0$ | Correlation Coefficient |
|--------|-------------------------|-------------|-------------|---------|-------------------------|
| US2-1  | 1.609                   | 0.28        | 0.56        | 0.60    | 0.93                    |
| US2-2  | 1.581                   | 0.26        | 0.46        | 0.55    | 0.95                    |
| US2-3  | 1.664                   | 0.28        | 0.55        | 0.59    | 0.96                    |
| US3-1  | 1.763                   | 0.51        | 0.81        | 0.50    | 0.94                    |
| US3-2  | 1.709                   | 0.42        | 0.82        | 0.60    | 0.98                    |
| US3-3  | 1.737                   | 0.15        | 0.80        | 0.85    | 0.98                    |
| US4-1  | 1.874                   | 0.32        | 0.78        | 0.68    | 0.97                    |
| US4-2  | 1.792                   | 0.23        | 0.80        | 0.78    | 0.98                    |
| US4-3  | 1.804                   | 0.25        | 0.85        | 0.76    | 0.98                    |
| US5-1  | 1.976                   | 0.30        | 0.85        | 0.72    | 0.96                    |
| US5-2  | 1.895                   | 0.35        | 0.83        | 0.68    | 0.94                    |
| US5-3  | 1.983                   | 0.34        | 0.81        | 0.66    | 0.95                    |
| US6-1  | 2.113                   | 0.41        | 0.81        | 0.60    | 0.93                    |
| US6-2  | 2.034                   | 0.40        | 0.80        | 0.60    | 0.95                    |
| US6-3  | 2.164                   | 0.21        | 0.85        | 0.82    | 0.99                    |

As shown in Figure 12, the modified damage constitutive relationship (Equation (25)), considering the chemical-stress coupling factor  $\mu$ , is in good agreement with the test results and the change of compaction stage can be well described. In addition, the experimental data of Gu [10] were applied to verify the effectiveness of the modified damage constitutive model. The mechanical response of coal under different soaking times was analyzed in Gu's study. Figure 13 displays the verification of the modified damage constitutive model with the experimental data of Gu. It is obvious that the modified model is highly consistent with the test curve. The modified model well described the stress–strain curves of coal under the different soaking times.

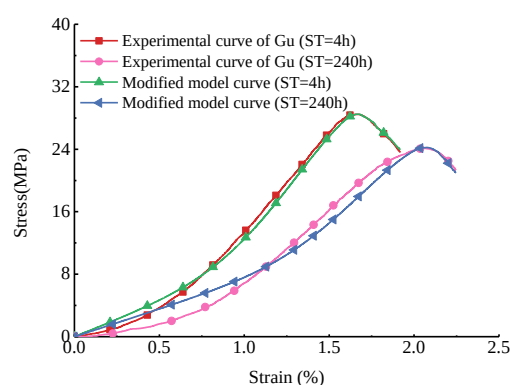


**Figure 13.** Verification of modified damage constitutive model with experimental data of Gu [10].

## 5. Conclusions

In this paper, physical and mechanical tests were carried out on coal samples subjected to a salty solution. The degradation mechanisms of the physical and mechanical properties of the coal samples were studied. A damage constitutive model for coal subjected to the salty solution under uniaxial compression was proposed by introducing the chemical-stress coupling factor. The relevant results are summarized as follows:

(1) The physical characteristics of coal under the action of salty solution show varying degrees of deterioration. Under the attack of salty solution, the relative mass difference of the coal samples displays an increasing trend at the beginning of the test, then gradually decreases. With the increase in immersion time, the pH value gradually tends to

be neutral. The mineral particles of coal samples are dissolved by the salty solution, resulting in aggravated chemical damage to the microstructure and an increase in the coal sample's porosity.

(2) The microstructure morphology of coal samples for different immersion times were analyzed. With the effect of the salty solution, the microscopic morphology has changed to a certain extent. As the immersion time increases, the number of mineral particles on the surface of the coal decreases significantly and the connectivity of the internal structure increases.

(3) The peak strength and elastic modulus of the coal samples changes exponentially as a function of soaking time. The failure strain exhibits an increasing trend with soaking time. As the soaking time increases, the initial damage of the coal sample increases exponentially. According to the theory of damage mechanics, the chemical-stress coupling damage variable was introduced, and the variation law of the coupled damage variable with time was obtained. Under the effect of salty solution, the changes of the stress–strain curve can be well described by modified damage constitutive model that considers the coupling factor  $\mu$ .

In the present study, the deterioration mechanism of coal under the action of salty solution was studied and a damage model for coal subjected to the salty solution under uniaxial compression was proposed. The modified damage model can well describe the changes in the compaction stage of the stress–strain curve caused by the water–rock reaction. The pre-peak behavior can also be well described by the modified model. However, the post-peak behavior cannot be fully characterized. In order to fully describe the mechanical behavior during rock failure, it is necessary to characterize the post-peak behavior. We will focus on this issue in future work.

**Author Contributions:** Conceptualization, methodology, formal analysis, software, validation, writing—original draft preparation, M.W.; writing—review and editing, Q.G.; supervision, project administration, Y.T.; funding acquisition, Q.G. and B.D. All authors have read and agreed to the published version of the manuscript.

**Funding:** This work was supported by the Open Fund of the Key Laboratory of Western Mine Exploitation and Hazard Prevention, Ministry of Education (SKLCRKF1901), Open Fund of State Key Laboratory of Water Resource Protection and Utilization in Coal Mining (grant no. GJNY-18-73.11), the National Natural Science Foundation of China (grant no. 51804163).

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** Not applicable.

**Conflicts of Interest:** The authors declare no conflict of interest.

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