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To cite this article: Lei Zhang *et al* 2019 *IOP Conf. Ser.: Earth Environ. Sci.* **267** 062025

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Experimental Study on Disintegration Characteristics of Soft Rock under Acidic Environment

Lei Zhang¹, Weike Li^{2,3*}, Janguang Huang², Huaguo Lin⁴

¹Guangdong University of Petrochemical Technology, Maoming, Guangdong Province, 525000, China

²Guangzhou Design Institute, Guangzhou, Guangdong Province, 510620, China

³Guangzhou University, Guangzhou, Guangdong Province,

⁴Guangdong Engineering Investigation Institute, Guangzhou, Guangdong Province, 510510, China

*Corresponding author's email: liweike@gzdi.com

Abstract. The Red-bed soft rock is widely distributed in South China, and it has been exposed to acid rain for a long time, which brings great risks to the stability of the engineering. Therefore, the Red-bed soft rock in South China is selected as the research object. A disintegration test under acidic environment was carried out, and the law of grain size change during disintegration is obtained. The research shows that: (1) Disintegration is a process in which large particles gradually transform into small particles; (2) Acidic environment promotes the disintegration of Red-bed soft rock, and the stronger the acidity, the stronger the disintegration; (3) In acidic environment, the disintegration of Red-bed soft rock can be divided into three stages: water absorption stage, rapid disintegration stage and slow disintegration stage; (4) Red-bed soft rock disintegration to different acidic solutions is different, which HNO₃ dominates the disintegration than H₂SO₄; (5) Due to the dissolution of mud, the main minerals participate in the reaction, causing rock cementation loss with pores larger, and finally lead to the softening and disintegration of the Red-bed soft rock.

1. Introduction

The acid rain is widely distributed in South China (Zou Delong, 2012) ^[1], where the PH value of acid rain solution is generally lower than 5.0 and even locally lower than 4.5. With the region where the acid rain is distributed, the soft rock solution will have stronger corrosion (Ou Yangjie, 2011) ^[2]. Therefore, the water-chemical-stress effect of the rock is obvious within the region where the acid rain is distributed (Shen Jingyi, 2012) ^[3]. As one class of representative soft rocks in South China, the "red-bed soft rock" has the following significant characteristics: the stress-release crack is developed, while it is softened in contact with the water, and quickly disintegrated, because the expansive minerals, clay minerals, soluble minerals, or organic components are rich in the soft rock, which is much easier for itself to have the volumetric expansion and dramatic strength reduction. Meanwhile, under the stress effect resulted from the acid solution environment and load, such process will also be superposed with the corrosion and damage effect of the acid substances upon some minerals contained in the soft rock, and the damage effect resulted from the stress concentration, which causes the soft rock to be subject to the catastrophic structural damage, and also causes the long-term stability of the engineering to be subject to the enormous risks. In previous studies, the greater significance was attached to the



hydrolysis effect (M. Iwata., 2011 ^[4]; Zhang Wei, Shang Yanjun, et al., 2013 ^[5]), the creepage/rheologic behavior (Zhu Jie, Xu Ying, et al., 2013 ^[6]), and the engineering support technology. However, less significance was attached to the researches on its disintegration process and damage process under the chemical-stress coupling conditions under the acid solution environment, which was perhaps one of the significant reasons for instability of soft rock side slope and tunnel's surrounding rock within the region where the acid rain was distributed. Therefore, this paper carries out the researches on the disintegration process and damage behavior of the soft rock under the acid solution environment, so as to understand the damage mechanism of the soft rock under the acid conditions and provide the references for the construction of soft rock engineering within the acid rain region.

2. Experimental Materials & Methods

2.1 Experimental materials

This experiment selects the soft rock samples taken from a given soft rock engineering in South China, which is the Neogene red-bed soft rock, featured in the clear bedding, low weathering degree, calcareous-argillaceous-cement-dominated, and good cementation. As shown by the testing results, its density is 2.30g/cm³. The main mineral components include the quartz, feldspar, calcite and a small amount of clay minerals.

2.2 Experimental ideas

For the properly prepared sample of red-bed soft rock, it is required to firstly carry out the acid solution test, then screen the sample soaked in the acid solution, and finally obtain each component mass through weighing. On this basis, this study adopts the fractal geometry theory to study the particle mass, and particle size fractal & morphological characteristics during the sample disintegration process. Meanwhile, this study also intends to study the variation regularity of ion concentration under the soaking environment of acid solution, and finally observe its morphological characteristics through scanning the electron microscope experiments.

2.3 Experimental methods

Before the soft rock disintegration experiment, it is required to weigh the rock sample mass and select the rock samples with the same or similar mass as the experimental object, so as to eliminate the mass impact upon the disintegration. It is required to prepare the acid solution as per a given proportion of H₂SO₄ & HNO₃, to form the solution with variable PH value and different proportion, as shown in Table 1.

Table1. Acid solution grouping

| H ₂ SO ₄ /HNO ₃ | pH | | | | |
|--|-----|-----|-----|-----|--------|
| | 3.0 | 4.0 | 5.0 | 5.5 | 7.0(J) |
| 0.7 | E | - | - | - | - |
| 2.5 | A | B | C | D | - |

Step 1: it is required to divide the acquired soft rock samples into 18 parts, separate each part of soft rock sample with the geotechnical sieve, and weigh the soft rock particles of variable grain sizes with the weighing scale, and obtain and record the initial proportion details of the variable-size particles in each part of the soft rock sample.

Step 2: It is required to separately soak the experimental samples with the properly prepared acid solution and distilled water (there are 3 experimental groups labeled as A, B, & C respectively, while each group contains 6 parts of rock samples, which will be simultaneously soaked, so as to conduct the concurrent experiment, reduce the experimental errors.) for 15 days. During such period, it is required to periodically measure the concentration and PH values of each ion contained in these two solutions, after the soaking. After 15 days, it is required to separately filter each part of sample with the filter

paper, so as to obtain the solid particles of the soft rock after the first soaking. Then, it is required to place the solid particles of the soft rock inside the drying oven for drying. After drying, it is required to separate each part of soft rock samples with the geotechnical sieve with the variable diameters, such as 10, 5, 2.5, 1.25, 0.63, 0.315, 0.16 and 0.08mm, and finally weigh the residual particle mass on the sieve. It is required to weigh the soft rock particles of variable grain sizes with the weighing scale, and obtain and record the first disintegration proportion details of the variable-size particles in each part of the soft rock sample.

Step 3: It is required to further subdivide the 6 parts of rock samples in each group into two subgroups, respectively with the serial numbers of A-1, A-2, B-1, B-2, C-1 & C-2. Then, it is required to continue to utilize the solution that has already soaked the samples at step 2 to soak the samples in subgroups A-1, B-1 & C-1, and utilize the properly prepared initial acid solution to soak the samples in subgroups A-2, B-2 & C-2, while each part of samples will be soaked for 15 days. During such period, it is required to periodically measure the concentration and PH values of each sort of ion inside the latter two sorts of solutions. After 15 days, it is required to separately filter each part of sample with the filter paper, so as to obtain the solid particles of the soft rock after the second soaking. Then, it is required to place the solid particles of the soft rock inside the drying oven for drying. After drying, it is required to separate each part of soft rock samples with the geotechnical sieve, and weigh the soft rock particles of variable grain sizes with the weighing scale, so as to obtain and record the second disintegration proportion details of the variable-size particles in each part of the soft rock sample. Then, it is required to repeat the aforesaid step 3 to carry out the experiment, until the proportion of variable-size particles in each part of the soft rock sample almost has no change.

3. Analysis of experimental results

3.1 Analysis methods

3.1.1 Disintegration rate theory

The disintegration behavior of the soft rock refers to the phenomenon that the water absorption & dispersion, and disintegration into broken pieces and particles will occur to given mass of soft rock under variable solution environments. The disintegration behavior of the soft rock will be quantified as per the soft rock disintegration rate α . The lower the disintegration rate is, the weaker the anti-scourability of the soil becomes. Vice versa. The higher the disintegration rate is, the stronger the anti-scourability of the soil becomes.

$$\alpha = |M1 - M2| / M0 \quad (1)$$

Where: M1 refers to the soft rock particle mass of a given sieve diameter under the designated grading time; M2 refers to the soft rock particle mass of a given sieve diameter under the next designated grading time; M0 refers to the total mass of soft rock before being soaked in the solution.

3.1.2 Fractal theory

When dealing with the distribution data of a large quantity of particles, the fractal and fractal dimension theory is introduced into the geotechnical engineering, while the fractal dimension will be used to characterize the relationship between the rock particle mass and grain size. Tyler & Wheatcraft gives the calculation model of grain size fractal dimension [7]:

$$\frac{M(r < R)}{M_T} = \left(\frac{R}{R_T} \right)^{3-D} \quad (2)$$

In equation (1), M_T refers to the mass of the particles with the size of less than R; refers to the total mass of the particles; R_T refers to the upper limit value of the particle size; D refers to the fractal dimension.

For the calculation of fractal dimension, it is required to take the logarithm for both sides of the equation (1), which will provide the linear representation regarding the fractal dimension D . Finally, it is required to calculate the value of D through the linear regression. Through the analysis & statistics of the experimental results, the D value under the variable acid environments will be obtained respectively.

3.2 Soft rock particle grading feature

During the process of disintegration experiment, the soft rock's particle grading will have the change of grading & morphological characteristics, with the increase of disintegration grading times. The particles with the larger grain size ($>10\text{mm}$) are obviously decreased, while the proportion of the particles with the smaller grain size are slightly and gradually increased. During the process of this experiment, this experiment adopts five comparison experiments, which enhances the comparability of the experiment and the accuracy of the results. During the disintegration process, we conduct the statistics & analysis upon the particle grain size, so as to try to eliminate the experimental errors and make the results have the comparability and implement the normalization & non-dimensionalization upon the experimental results. Finally, the particle distribution details after 10 times of disintegration are obtained. Due to the large quantity and length limitation, this paper only selects the PH and S/N (the ratio of sulphuric acid and nitrating acid in the solution) values of three variable solutions for analysis, while the results are as shown in Figure 1.

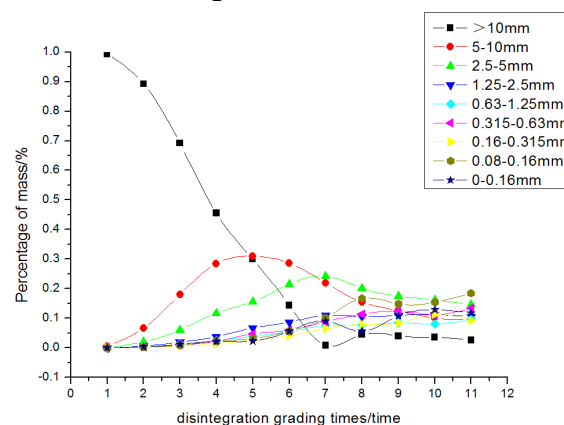


Figure1. Variation of particle distribution of soft rock disintegrated in acidic solution S/N=2.5 pH=3

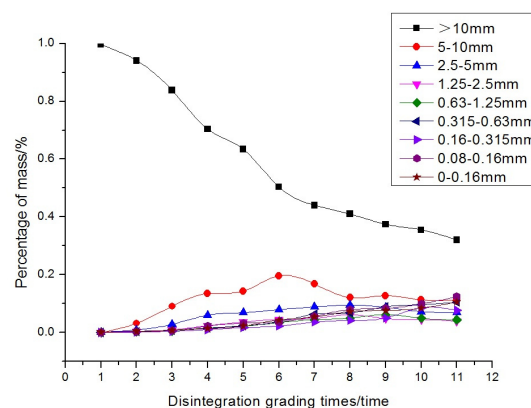


Figure2. Variation of particle distribution of soft rock disintegrated in acidic solution S/N=0.7 pH=3

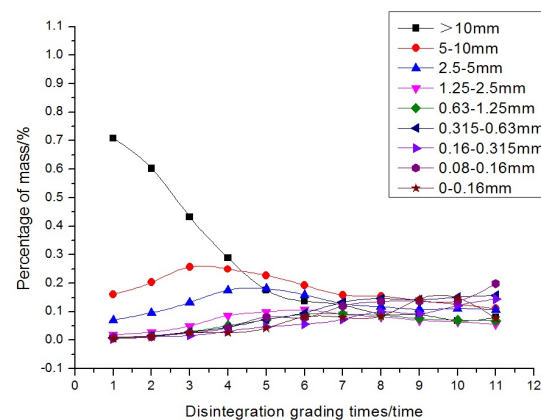


Figure3. Variation of particle distribution of soft rock disintegrated in acidic solution S/N=4 pH=3

As obviously shown from the aforesaid three groups of images, the particles with the larger grain size are obviously decreased, while the proportion of the particles with the smaller grain size are slightly increased. Besides, the percentage variation of particle distribution for particles with variable grain size also has the obvious difference. However, under the acid environment, the particles with the obvious different grain sizes generally make the particle grading become more and more even. Through describing the percentage variation laws of the particles with the specific grain size, the disintegration characteristics of the red-bed soft rock in the variable solutions may be obtained.

When handling the distribution data of a great number of particles, this subject experiment introduces the concept theory of disintegration rate. Through measuring the soft rock mass in a given diameter sieve (for instance, 10mm) under the initial grading status, and the soft rock mass in such sieve under each following grading status, it is required to adopt the following equation to demonstrate that it is a significant index to study the soft rock disintegration characteristics. Through studying the impact of PH and S/N values in the solution upon the soft rock disintegration rate, it is found that the larger the disintegration rate is, the more obvious the effect of soft rock disintegration becomes.

Table2. Fractural dimension of soft rock disintegration under different acidic environments

| Types of solution Disintegration rate | | Soaking times | | | | | | | | | |
|--|-------------------|---------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| | | Initial | 1 st | 2 nd | 3 rd | 4 th | 5 th | 6 th | 7 th | 8 th | 9 th |
| 1 | S/N=2.5 pH=3 | 0.172 | 0.293 | 0.587 | 0.676 | 0.758 | 0.765 | 0.779 | 0.778 | 0.802 | 0.805 |
| 2 | S/N=2.5 pH=4 | 0.107 | 0.308 | 0.545 | 0.702 | 0.856 | 0.992 | 0.955 | 0.960 | 0.964 | 0.975 |
| 3 | S/N=2.5 pH=5 | 0.076 | 0.226 | 0.383 | 0.468 | 0.531 | 0.547 | 0.568 | 0.584 | 0.587 | 0.913 |
| 4 | S/N=2.5 pH=5.5 | 0.094 | 0.311 | 0.560 | 0.711 | 0.762 | 0.797 | 0.852 | 0.892 | 0.905 | 0.912 |
| 5 | S/N=0.7 pH=3 | 0.058 | 0.163 | 0.297 | 0.369 | 0.502 | 0.565 | 0.597 | 0.633 | 0.655 | 0.688 |

| | | | | | | | | | | | |
|----|-----------------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
| 6 | S/N=4 pH=3 | 0.151 | 0.393 | 0.597 | 0.755 | 0.811 | 0.830 | 0.873 | 0.878 | 0.907 | 0.892 |
| 7 | S/N=4 pH=4 | 0.085 | 0.258 | 0.484 | 0.590 | 0.643 | 0.650 | 0.655 | 0.671 | 0.684 | 0.694 |
| 8 | S/N=4 pH=5 | 0.222 | 0.425 | 0.634 | 0.718 | 0.760 | 0.770 | 0.779 | 0.779 | 0.801 | 0.801 |
| 9 | S/N=4 pH=5.5 | 0.092 | 0.253 | 0.461 | 0.587 | 0.625 | 0.652 | 0.664 | 0.681 | 0.682 | 0.701 |
| 10 | pH=7 | 0.182 | 0.384 | 0.468 | 0.614 | 0.684 | 0.679 | 0.738 | 0.749 | 0.778 | 0.788 |

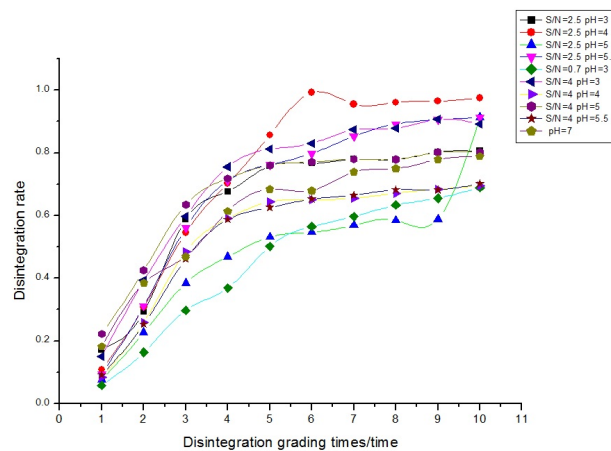


Figure4. Fractal dimension curve of disintegration of Red-bed soft rock in different solutions

The curve of disintegration rate reflects the rate at which the rock will be disintegrated into the even particles under the acid solution conditions. The larger disintegration rate is, the more dispersed the rock particle become. This paper uses the disintegration rate to characterize the disintegration conditions of the soft rock under the acid solution environment.

In order to characterize the impact laws of variable acid environment upon the disintegration effect of the red-bed soft rock, the fractal dimension variation figure (Figure 2) under the variable acid solutions conditions are respectively drawn. As shown from Figure 2, in the pure water, the fractal dimension of the soft rock is the lowest, which indicates that the acid environment has the obvious promotion function upon the disintegration of red-bed soft rock. Besides, with the increase of soaking times, the fractal dimension gradually increases, while the disintegration of soft rock is a process developed from 2D to 3D.

The disintegration of red-bed soft rock is a non-linear process, which will make the numerical fitting of all curves, which may provide the relational expression of the fractal dimension D and soaking times n during the disintegration process:

$$D = a \cdot e^{b \cdot n} + c \quad (2)$$

Such expression may semi-quantitatively reflect the disintegration rate of the soft rock in the variable solutions.

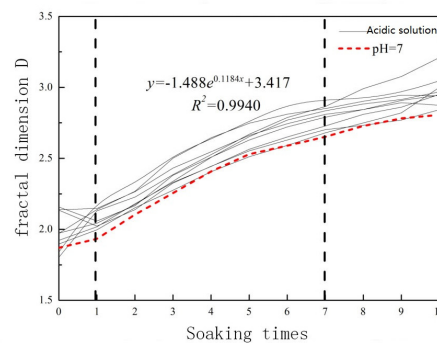


Figure 5. Fractal dimension curve of disintegration of Red-bed soft rock in different solutions

Through the comparison between the disintegration rate curve and fractal dimension variation curve, it is found that great similarity exists between the variation trends of both of them. Combined with these two curves, it is found that the disintegration of soft rock has obvious stage features. At the stage where the disintegration starts is the rapid disintegration stage. At such stage, the rock starts the disintegration, while the slope is also the largest and the disintegration rate is the fastest. In the experiment, a lot of loosening and disintegration of the rock sample is observed. During such process, the content of acid ion in the solution is much larger. Therefore, it has much larger impact upon the disintegration of the soft rock, which causes the disintegration rate to be the fastest. After this, it will be the slow integration or stable integration stage. The disintegration rate will slow down, while most of the rocks are disintegrated, and the residuals are some mineral components that are difficult to be soluble in water and hard to participate in the reaction. As shown from the curve of disintegration rate, it is found that under the environment of solutions with the variable PH values, the disintegration rate of soft rock also varies. When the PH value is 3 & 4, the slope of the curve is the largest, which indicates that the disintegration rate is the fastest. However, under the same PH value conditions, the different S/N values will also have the variable disintegration rates of the soft rock. Under the identical PH value zone, the larger the S/N becomes, the slower disintegration rate of the soft rock becomes, which indicates that the impact of HNO_3 is much larger than that of H_2SO_4 during the disintegration process of the soft rock.

In summary, it is found that the acid solution has the obvious promotion function upon the disintegration of soft rock. Besides, the stronger the acid is, the faster the disintegration becomes.

Meanwhile, the impact of HNO_3 is much larger than that of H_2SO_4 during the disintegration process of soft rock.

4. Conclusion

Through the experimental research upon the soft rock disintegration behavior under the variable acid environments, the following several findings are obtained:

(1) All the soft rocks will be disintegrated in the acid solution. The acid solution has the obvious promotion functions upon the soft rock disintegration. Besides, the stronger the acid is, the stronger the disintegration becomes.

(2) Under the variable acid solution environments, the soft rock will have variable disintegration rate. Among them, the stronger the acid is, the quicker the disintegration rate of the soft rock becomes. Among them, as shown by the experimental data, when the PH value is 3, the slope of the disintegration curve is the largest, which indicates that the disintegration rate is the largest.

(4) Under the same PH value, the variable S/N value will have different disintegration rate of the soft rock. Within the zone of the same PH value, the larger the ratio of H_2SO_4 & HNO_3 is, the slower the disintegration rate of the soft rock becomes, which indicates that during the disintegration process of the soft rock, the effect of HNO_3 is much stronger than that of H_2SO_4 .

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