

Laboratory weathering experiment on sandstone of Niche of Sakyamuni Entering Nirvana at Dazu Rock Carvings, China

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May 5, 2020

Abstract

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1.0 Title

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2.0 Abstract

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is granular disintegration. For all experiments, the variation in mass decayed exponentially with the periods; while the variation in surface hardness and P-wave velocity decayed as a power function with the periods. The rankings of influencing environmental factors of sandstone weathering follow this order: acid rain cycle, freeze-thaw cycle, and dry-wet cycle. More generally, the water-rock interaction (WRI) occurs from the surface to the inner structure of the sandstone. Granular disintegration is driven by (1) the dissolution of calcite, alteration of feldspar, and water swelling and drying shrinkage of smectite; and (2) the widening inter-granular micro-cracks. The findings could provide useful insights for the protection of the studied statues.

3.0 Keywords

sandstone, Niche of Sakyamuni Entering Nirvana, dry-wet cycle, freeze-thaw cycle, acid rain cycle

4.0 Introduction

Dazu Rock Carvings can be perceived as an example of the highest level of Chinese cave temple art from the 9th to 13th centuries. The United Nations Educational, Scientific and Cultural Organization (UNESCO) designed them as the world cultural heritage in December 1999. In particular, the Niche of Sakyamuni Entering Nirvana constructed of sandstone is the largest bust of Sakyamuni in the world, and also an important part of Dazu Rock Carvings (Tong, 2003). It has suffered from extensive surface weathering over the past 1000 years. The surface weathering diseases include efflorescence, granular disintegration, biological colonization, cracks, seepage, scaling, blistering and missing parts, and granular disintegration in total dominates that. Therefore, the formation and mechanism of granular disintegration is the research object of the studied statues.

Until lately, the study on granular disintegration adopts on-site investigation and description as the key study approach. Traditionally, the development of granular disintegration on rock surfaces can be attributed to physical weathering such as dry-wet cycle; freeze-thaw cycle and salt crystallization due to water absorption and temperature (Rodriguez-Navarro, Linares-Fernandez, Doehe, & Sebastian, 2002; Cardell et al., 2003; Oguchi & Yuasa, 2010; Labus & Bochen, 2012); and chemical action including dissolution, oxidation, reduction and hydrolysis (Prieto, Silva, Rivas, Wierchos, & Ascaso, 1997; Pera, Arribas, Critelli, & Tortosa, 2001; Herrera, Arroyave, Guimet, de Saravia, & Videla, 2004; Lee, Jo, & Kim, 2011). However, Ilies, Irimuş, and Rus (2015) pointed out that over-reliance on field observations limited our understanding of the granular disintegration formation. Recognition of this limitation is implicit in many studies which sought to determine physical and mechanical parameters from the macro level, including, e.g., mechanical strength, porosity, ultrasonic velocity, mass and water absorption (Jeng, Lin, & Huang, 2000; Tan, Chen, Yang, & Cao, 2011; Özbek, 2014). Nonetheless, few micro level studies were conducted using indirect methods, e.g., XRD, SEM and chemical analyses. They can be usefully complemented by (1) providing direct evidence of cracks and pores in rocks and their constituent grains; and (2) analyzing mineral and chemical composition to evaluate their potential contributions towards rock deterioration (Clément & Kimpe, 1977; Pye & Cambridge, 1985; Sassoni & Franzoni, 2014).

Since the 1990s, some studies have been conducted to understand the weathering effects on Niche of Sakyamuni Entering Nirvana. These studies include preserving environmental and meteorological characteristics (Zhou & Deng, 2002; Chen, Jiang, & Xi, 2004); effect of the water seepage (Wang & Zhang, 1992; Zhang & Jiang, 2016); hydraulic parameters and seepage model of rock (Wang, Wang, & Zhang, 1999; Wang, Wen, Wang, & Zhang, 2001); prevention of water hazard and destruction mechanism of Na₂SO₄ crystals on sandstone by laboratory accelerating ageing experiments (Tan, 2013); surface weathering characteristics and degree of sandstone (Zhang et al., 2018). However, the relation between granular disintegration and sandstone weathering mechanism has not been revealed experimentally.

In this study, we conducted three weathering experiments to examine different influences on sandstone weathering of Niche of Sakyamuni Entering Nirvana. From these experiments, we analyze the changes in physical and mechanical properties of sandstone and the sandstone weathering mechanism. The research outcomes could provide some physical and mechanical parameters and basis for the protection of the studied

statues.

5.0 Sample preparation and properties

5.1 Studied site

Niche of Sakyamuni Entering Nirvana is located in the Dazu District of Chongqing, China (Fig. 1). Its dimensions are 31.60 m in length, 4.46 m in depth, and 6.72 m in height (shown in Fig. 2.). It can be divided into three parts, i.e., upper, middle and lower parts. The Sleeping Buddha lies in the middle part. The lower part (i.e., below the Sleeping Buddha) consists of four Kings, the Emperor, and 13 Buddhas and Bodhisattvas, while the upper part (standing on the top of Sleeping Buddha) includes nine maid statues standing on clouds.

5.2 Sandstone ore appraisal

The sandstone ore appraisal is shown in Fig. 3. The sandstone is a stratum under the Pen-laizhen group of late Jurassic. The sandstone is of medium maturity and composed of detritus (84%) and fillings (16 %). The detritus covers 62% quartz (Q), 10% plagioclase (Pl), 8% K-feldspar (Kf), 2% mica, and 2% chlorite with good sorting and psephicity; while the fillings include 4% mud and 12% calcite. The grain-sizes are 0.08-0.25 mm.

5.3 Physical and mechanical properties

The physical and mechanical properties are evaluated in accordance to the ISRM standards. The results are listed in Table 1.

5.4 Sample preparation

Fig. 4 shows the detailed conditions of the geological body of Niche of Sakyamuni Entering Nirvana. It is divided (from top to bottom) into four strata, i.e., Layer A, Layer B, Layer C, and Layer D. Layer A and Layer B have similar compositions which are composed of medium-to fine-grained feldspar quartz sandstone. Layer A has the horizontal bedding structure and its bedding is developed, while Layer B has both cross-bedded and horizontal bedding structures and its bedding is not developed. There is a thin mudstone interlayer between Layer A and Layer B. Layer C is composed of fine-grained feldspar quartz sandstone which has horizontal bedding structure. Finally, Layer D consists of greyish brown silty mudstone (Zhang et al., 2018). In current study, the sandstone samples were collected from Layer A (called LA) and Layer B (called LB), which are in the core of the same layer of Niche of Sakyamuni Entering Nirvana. All core samples were of same orientation, and they were prepared as cylinders with a diameter of 50 mm and a height of 50 mm. The samples for each experiment are shown in Table 2.

6.0 Experimental process design

6.1 Experimental procedures

Based on the meteorological and environmental characteristics of Dazu Rock Carvings (Chen et al., 2004; Zhang, 2015), the highest and lowest monthly mean air temperatures for 1975-2011 were 39.5 (in August) and -3.4 (in December), respectively. The mean annual relative humidity was about 83%. The maximum and minimum monthly mean rainfalls were 490.7 mm (in July) and 54.5 mm (in December), respectively. The wet days ranged from 147 to 174 days, accounting for 2/5 to 1/2 of the days in a year. Besides, the proportion of acidic rainfall with pH<5.6 was 77.0 %, where almost of the rainfalls were acidic. In a sense, water is the main cause of sandstone weathering in Niche of Sakyamuni Entering Nirvana. For this reason, the study site might have exhibited three kinds of weathering processes: dry-wet cycle (DW), freeze-thaw cycle and acid rain cycle.

6.1.1 Dry-wet cycle

From the daily average humidity variation in Niche of Sakyamuni Entering Nirvana (Chen et al., 2004), the reduction time (9:00-21:00) of humidity and maintenance time (21:00-9:00) at high humidity in a day was

about 1:1. The sandstone samples were air-dried (9:00-21:00) and placed into a container filled with distilled water (21:00-9:00). This process represented the completion of a cycle, and seven cycles were set as one period, as shown in Fig. 5.

6.1.2 Freeze-thaw cycle

From the daily temperature variation in Niche of Sakyamuni Entering Nirvana (Chen et al., 2004), the temperature rose after 9:00; peaked around 15:00-18:00; gradually decreased, and stabilized about 22:00-9:00. As a result, the samples of freeze-thaw cycle were divided into two groups: saturated group (SG) and dry group (DG). All samples were oven-dried at 40 (9:00-19:00); the saturated group was saturated in the container (19:00-22:00), and the dry group was placed in the air; they were then placed in the freezer at -5 at 22:00-9:00. That corresponded to the completion of a cycle, and seven cycles were set as one period, as shown in Fig. 6.

6.1.3 Acid rain cycle

Since the acid at the study site is a mixture of sulfuric and nitric acid (Zhang, 2015), the acid rain cycle was set up in such a way that the sandstone samples were submerged in solutions with pH=2.6, pH=4.1 and pH=5.6, respectively. They were configured with dilute hydrochloric solution and distilled water; 0.009 g/L of sodium nitrate solution, 0.0285 g/L of potassium sulfate solution and 0.0160 g/L of ammonium nitrate solution were added into the solutions with different pH values. After that, the sandstone samples were submerged in these solutions overnight (21:00- to 21:00 on the next day). Then, they were air-dried for the subsequent 24 hours. This procedure was considered a cycle, and three cycles were set as one period. The pH value of solution was adjusted to original value with dilute hydrochloric solution before each soaking, as shown in Fig. 7.

6.2 Experimental parameters and analytical method

All samples were first dried at 105 for at least 48h, and cooled down until a constant mass was reached. After that, the mass, surface hardness and P-wave velocity were measured and the surface microstructure of samples was photographed at fixed points using a microscope. For each sample, all these parameters were re-determined after every period. At the end of the experiments, the XRD and SEM of samples were measured in order to analyze the sandstone weathering mechanism. Fig.8 shows that the sampling locations of XRD and SEM are the surface (red), middle (blue) and inner (green) parts. Three experiments were conducted in 50 periods that lasted for 350 days.

6.3 Test facilities

Regarding the test facilities, an electronic balance (Type JJ1000Y) was used for testing the mass of sample (accuracy 0.01g). A leeb hardness tester (Type TH-120A(D)) was applied for testing the surface strength of sample. A supersonic reflectoscope (Type RSM-SY5N) was utilized for testing the P-wave velocity of sample, a full set of touch-based sampling analysis software that can preset the lead time and find the initial value automatically. Portable micrograph (Type 3R-WMMOTV) was used for the surface micrograph of sample (resolution of 960*240). Refrigerating machine (Type DW-40) was adopted for freezing the sample (with a volume of 80L and a minimum temperature of -40). A dry oven (Type 101-3SB) was applied for thawing the sample (the maximum heating temperature can reach 300). A low vacuum scanning electron microscope (Type JSM-5600LV) was used for analyzing the microstructure of sample (HV resolution of 3.5nm, and LV resolution of 5.0nm.). A X-ray powder diffractometer (Type D/Max-TTRIII) was utilized for analyzing the mineral and chemical composition of the sample, sharp shadow high temperature in situ material structure analysis system and Galipixel 2d and Xe proportional detector.

7.0 Results

7.1 The deteriorated features

Samples as a result of 50 periods of three experiments were deteriorated (Figs. 9, 10, 11). The results demonstrate that the most severe deterioration was driven by acid rain cycle. With increasing periods,

many granules fell off and yellow spots were visible. By contrast, samples did not deteriorate in the dry-wet and freeze-thaw cycles (despite a small amount of granules fell off.). Hence one main type of failure is observed in all of the samples, i.e., granular disintegration.

7.2 Surface microstructure

The surface microstructure of samples at 60 times in three experiments was shown in Fig. 12, 13, 14. Quartz, feldspar and other grains of fresh sandstone were closely arranged. The grains were filled with white cement with clear boundary contour and smooth surface. As the experiment went on, all samples experienced granular disintegration. On the other hand, the surface color of samples turned dark and black spots were visible. Besides, a small number of pores and micro-cracks reflected that the structure got loose. Some yellow spots appeared on the samples under the effects of acid rain cycle. The deterioration of samples in acid rain solution (especially at higher H^+ concentrations) was more serious and rapid than that of samples affected by dry-wet and freeze-thaw cycles.

7.3 Mass, surface hardness and P-wave velocity test

The variations in mass, surface hardness and P-wave velocity with the increasing periods are normalized using the ratio of the value at the end of each period to the initial value (0 period). The fitting curves of these normalized values versus the number of periods are shown in Figs.15, 16, and 17, respectively.

7.3.1 Massvariation

Fig.15 indicates that the sample mass varies in the same manner under the effects of different experiments. The sample mass increased initially because each sample imbibed water at the early stage of the experiment (0-3 periods) and decreased subsequently. The sample mass decreases the most in response to the effects of acid rain cycle, followed by the saturated group. Whilst the deterioration of samples is less severe in the dry-wet cycle, it is greater than that of the dry group. Obviously, the mass deterioration of LA samples is faster than that of LB samples under the same experimental treatment.

7.3.2 Surface hardness and P-wave Velocity variations

The surface hardness (Fig. 16) and P-wave velocity (Fig. 17) decrease with increasing periods across all experiments. With an increasing number of periods, the surface hardness and P-wave velocity of samples gradually decrease, whereas the surface hardness decreases faster than the P-wave velocity implying that the deteriorations of samples start from the surface by water and transfer to the inner little. The surface hardness and P-wave velocity decreasing rate are the highest when the samples are immersed in $pH=2.6$; the lowest in the dry group; ranked in the middle in the dry-wet cycle and saturated group. The surface hardness and P-wave velocity deterioration of LA samples are greater than that of LB samples when other conditions remain the same.

7.3.3 The parameters loss rate

From Fig. 18, it can be seen that different experiments might have different effects on the deterioration to samples. The effect of acid rain cycle on the parameters loss rate of samples is greater than that of samples depending on the dry-wet cycle and freeze-thaw cycle, and the greatest in $pH=2.6$. The samples are also deteriorated by the dry-wet cycle and freeze-thaw cycle as well. Among them, the parameters loss rate of saturated group is higher than that of samples after the dry-wet cycle, and the lowest in dry group. The parameters loss rate of samples follows this order: $pH=2.6 > pH=4.1 > pH=5.6 > \text{saturated group} > \text{dry-wet cycle} > \text{dry group}$. This indicates that the samples are more susceptible to the effects of acid rain cycle. Furthermore, the drop in parameters of LA samples is larger than that of LB samples in different experiments, signifying that the sandstone of bedding development is among the first to be affected by weathering. The results suggest that the weathering degree of the upper part of Niche of Sakyamuni Entering Nirvana is more serious than that of the middle part accompanied by a large amount of granular disintegration.

7.3.4 Results of regression analyses

The equations and correlation coefficients obtained from the plots are given in Table 3. An exponential function is adopted to fit the variation in mass results:

$$m=1-aex/t \quad (1)$$

where m is the mass variation in different cycles; a and t are constants; x is the experimental period.

Meanwhile, a power function is adopted to fit the variations in surface hardness and P-wave velocity results:

$$H(V_p)=1-axc \quad (2)$$

where $H(V_p)$ is the variation in surface hardness (P-wave velocity) in different cycles; a and c are constants; x is the experimental period.

The correlation coefficients for mass, surface hardness and P-wave velocity versus the cycles of three experiments range between 0.95 and 0.98, suggesting that they can capture the changes of these parameters. All these results are basically consistent with Fang et al. (2015) and Ozbek (2014).

The variation in mass is only applicable to the sandstone weathering rate with cracks or flaking. The surface hardness and P-wave velocity of each sample gradually decrease with increasing cycles, which can be considered as the acceleration of sandstone weathering rate. It is thus limited to evaluate the weathering rate by calculating the variation in mass of sandstone, and the variation in surface hardness and P-wave velocity can be properly established to test the approximate value of the weathering rate (Li, Wang, & Chikaosa, 2008). The approximate value of sandstone weathering rate can be established using the following formula:

$$V = 1-A XC \quad (C>0) \quad (3)$$

where V is the approximate value of the weathering rate; A is the coefficient of weathering decline, i.e., the higher the coefficient of weathering decline is, the faster the weathering rate is; X is the weathering period; and C is a constant.

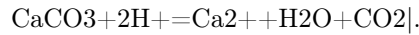
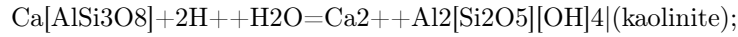
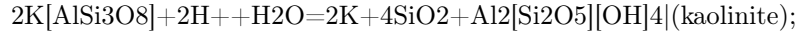
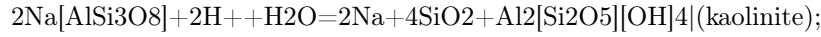
8.0 Analysis of sandstone weathering mechanism

8.1 Mineral and chemical composition analysis

The mineral and chemical composition of samples before and after the experiment is displayed in Figs. 19, 20, 21, Tables 4, 5, 6. The mineral composition of fresh sandstone comprises quartz, K-feldspar, plagioclase, clay minerals and calcite; and clay minerals comprise smectite, illite and chlorite. After the experiment, the results indicate that plagioclase, K-feldspar, calcite and clay minerals contents decreased. The drop in the mineral content of the samples is the lowest inside the sample; the highest on the surface; and in the middle beneath the sample surface. Since the sandstone surface is more prone to carry out WRI, and water acts on sandstone surface for a long time, while water infiltrates into the inner for a certain time. These caused the surface hardness to drop faster than the P-wave velocity. Calcite content decreases the most due to its instability, especially the surface calcite of pH=2.6 in the main disappears, demonstrating the weathering rate of calcite between grains is much higher than that of grains, and to slow down the sandstone weathering rate, the key is thus to control the dissolution of inter-granular material. Although dry smectite is stable in natural state, it often drives water swelling and drying shrinkage in response to the change in humidity, hence causing granular disintegration (Li & Chikaosa, 2005). The chemical properties of quartz are stable with strong weathering resistance, resulting in the increase of quartz after the experiment.

There are no new minerals generated in the dry-wet and freeze-thaw cycles. We deduce that the dry-wet cycle and freeze-thaw cycle on the deterioration of sandstone are physical deterioration. In fact, mineral grains and cement of sandstone are continuously lubricated and softened with the increasing dry-wet cycles (Sumner & Loubser, 2010). Furthermore, the water stored in the pores of sandstone freezes and expands under freeze-thaw cycles, causing tensile stress and micro-cracks in the sandstone. When the pore water or crack water melts, water enters into the micro-pores and micro-cracks inside the sandstone if the temperature alternates positively and negatively (Tan et al., 2011). Nevertheless, the chemical reaction between calcite and acid rain solution reduces the content of CaCO_3 . Other minerals that are non-reactive to corrosion

are enriched on the surface of the samples which means could change the color of the samples. This might explain the occurrence of yellow spots on the samples (Sun, Li, Zhang, Wang, & Wang, 2010). Kaolinite, a new mineral, is produced on the sandstone after acid rain cycle. The main reason is that plagioclase and K-feldspar are more unstable. The following chemical reactions are likely to occur (Lee et al., 2011):



Besides, chemical substances dissolved in acid rain solution would be taken away removed from the sandstone surface, and acid rain cycle is affected by the effects of dry-wet cycle which would accelerate chemical corrosion inside the sandstone.

8.2 Microstructure analysis

The SEM images at 2000 times (shown in Figs. 22, 23 and 24) reveal the microstructure of samples. The structure of fresh sandstone is characterized by its good homogeneity and compactness, but without apparent layered phenomenon. There are some clay minerals containing illite, smectite and chlorite. After the experiment, the sandstone starts to show obvious changes in its morphology from the surface to the inner structure. The surface structure deterioration is characterized by huge calcite loss, and secondary crack along the grain boundary, resulting in a loose and porous microstructure. The deterioration is particularly serious with corrosion holes in the acid rain cycle. The middle and inner structure deterioration is relatively minor, and the minerals between grain boundaries drift away due to reactions of water, generating pores and micro-cracks. There are a few clay minerals around the pores and micro-cracks, e.g., kaolinite, chlorite, illite, smectite, illite/smectite formation and chlorite/smectite formation. To sum up, the main mechanism involved in granular disintegration appears to be gradual widening of grain boundary micro-cracks and, to a lesser extent, development of other micro-cracks. The effects are: (1) it diminishes the degree to which the grains in the sandstone interlock, as reducing the mass, surface hardness and P-wave velocity; and (2) it facilitates the ingress of water, thereby accelerating the weathering rate (Sassoni & Franzoni, 2014).

By comparing the mineral and chemical composition and microstructure analysis of sandstone, we find that the surface deterioration of sandstone is more severe. This means that the WRI occurred from the surface to the inner structure. The statues in Niche of Sakyamuni Entering Nirvana are in the open air, hence the deterioration by water (including precipitation and capillary water) is serious and irreversible.

9.0 Conclusions

The physical and mechanical properties of sandstone (sampled from Niche of Sakyamuni Entering Nirvana) as a function of dry-wet cycle, freeze-thaw cycle and acid rain cycle were weakened using a series of tests on a macro-scale. The mineral, chemical compositions and microstructure deteriorated on a micro-scale. The conclusions are:

- (1) The main deterioration is granular disintegration under different weathering conditions. The sandstone weathering is more significant on sandstone with bedding development, and the weathering rate escalates with various numbers of cycles.
- (2) The variation in mass is attenuated by an exponential function, while the variations in surface hardness and P-wave velocity are attenuated by a power function. Besides, the approximate value of weathering rate is derived from the variation in surface hardness and P-wave velocity.
- (3) The deterioration degrees affecting the sandstone weathering follow this order: acid rain cycle, freeze-thaw cycle, dry-wet cycle. Notably, higher H^+ concentrations would enhance the sandstone weathering. To control the harm of acid rain, antiseptic materials should be applied on the statue surface.

(4) The deterioration of sandstone by water occurs from the surface to the inner structure. The surface structure of sandstone is the most susceptible to weathering effect. The dissolution of calcareous cement, alteration of feldspar, and water swelling and drying shrinkage of smectite on account of moisture change make the sandstone surface loose, and the grains are taken away by water, leading to the granular disintegration. SEM demonstrates that granular disintegration is primarily driven by the widening inter-granular micro-cracks.

(5) In this paper, the weathering effects of sandstone of Niche of Sakyamuni Entering Nirvana were studied experimentally. However, laboratory experiments were limited by time period, and the sandstone weathering effects of studied statues is longer and more intense. The research is thus intended to experiment for a long time.

10.0 Acknowledgments

Funded by Sichuan and Chongqing Grottoes protection demonstration project (Dazu Rock Carving Niche of Sakyamuni Entering Nirvana and Small Fowan statues conservation and restoration project) undertaken by the Chinese Academy of Cultural Heritage, as well as by the basic scientific research service fee of the Chinese Academy of Cultural Heritage (2016-JBKY-06).

11.0 Data Availability Statement The data that support the findings of this study are available from the corresponding author upon reasonable request.

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