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Laboratory test on moisture adsorption-desorption of wall paintings at Mogao Grottoes, China*

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Abstract: Moisture adsorption-desorption tests (MATs) were conducted on simulated mural plaster specimens under different air temperatures (ATs) and relative humidity (RH) to investigate the possible effect of seasonal alteration and visitors' breath on the deterioration of Mogao Grottoes, Dunhuang, China. Saturated salt solutions were used to maintain a constant RH, and plant growth cabinets were used to maintain a constant or varying temperature in the simulation test. The weight of specimen was periodically measured to determine the adsorbed or desorbed moisture. Test data illustrate that the desorption process is far quicker than the adsorption one, indicating that it is possible to inhibit the disadvantage effect from visitors, such as shortening the staying time in caves. In case of high humidity, an accumulated moisture adsorption was found to correspond to the varying temperature. On the other hand, in case of low humidity, accumulated moisture desorption corresponded. Test data imply that opening caves more often to visitors in humid seasons should be avoided so as to prevent continuous wetting of wall paintings.

Key words: Mogao Grottoes, Wall paintings, Plaster, Moisture adsorption-desorption, Unsaturated soil
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1 Introduction

Dunhuang is a strategically located early Chinese settlement far beyond western limit and at the point where the two branches of the Silk Road converge. The Mogao Grottoes were excavated into the conglomerate cliff face (Fig. 1) at the eastern foot of the Mingsha Mountain in Dunhuang City, northwest of China. The Mogao Grottoes are famous for wall paintings, painted sculptures and wooden cave temple fronts, etc. The Grottoes have a history of more than 1600 years, of which some caves were first built in 336 AD and later became a huge group of caves with rich contents after construction via 16 Kingdoms. Due to extremely valuable wall paintings, sculptures and the profound and diversified Buddhist art, the Grottoes were listed as a World Cultural Heritage Site by the United Nations Educational, Scientific and Cultural Organization (UNESCO) in 1987. There are 750 caves consisting of five tiers from the top to the bottom, stretching about 1600 m long in the precipice body (Fig. 1a), and 45 000 m^2 wall paintings have been preserved (Guo *et al.*, 2009). The vulnerable wall paintings suffered various kinds of damages caused by different microenvironments (Miura *et al.*, 1990; Li, 2010). Previous research proved that the wall paintings damages result from efflorescence of soluble salts such as halite because of moisture infiltration and migration. Moisture moves through the plaster layer of wall paintings, and then evaporates

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from the surface. Seasonal air circulation in the grottoes probably accelerates evaporation. This process causes flaking of the mural influenced by efflorescence of soluble salts (Torraca, 1983; Miura *et al*., 1997). Also, visitors' breath can result in deterioration (Zhang *et al*., 2005; Altenburg *et al*., 2010). Obvious alterations were recorded in relative humidity (RH), temperature, and carbon dioxide after visitors' entering caves. The respiration disturbed the environmental balance and caused damage to the wall paintings preservation (Maekawa *et al*., 1997; Demas *et al*., 2010). Salt efflorescence (Fig. 2), one of the typical diseases produced by moisture migration, is characterized by the granular desegregations and crumbling of soil materials (Wilson-Yang and George, 1989). With further development of salt diseases, the plaster of wall paintings become dilapidated and the paint layer shows obscission due to salt heaving (Zhang M.Q. *et al*., 1995; Zhang H.Y. *et al*., 2008).

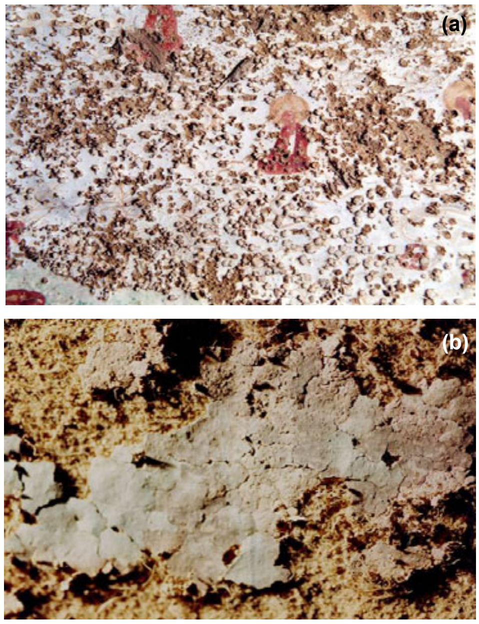


Fig. 2 Salt blister (a) and disruption (b) of wall paintings at Mogao Grottoes

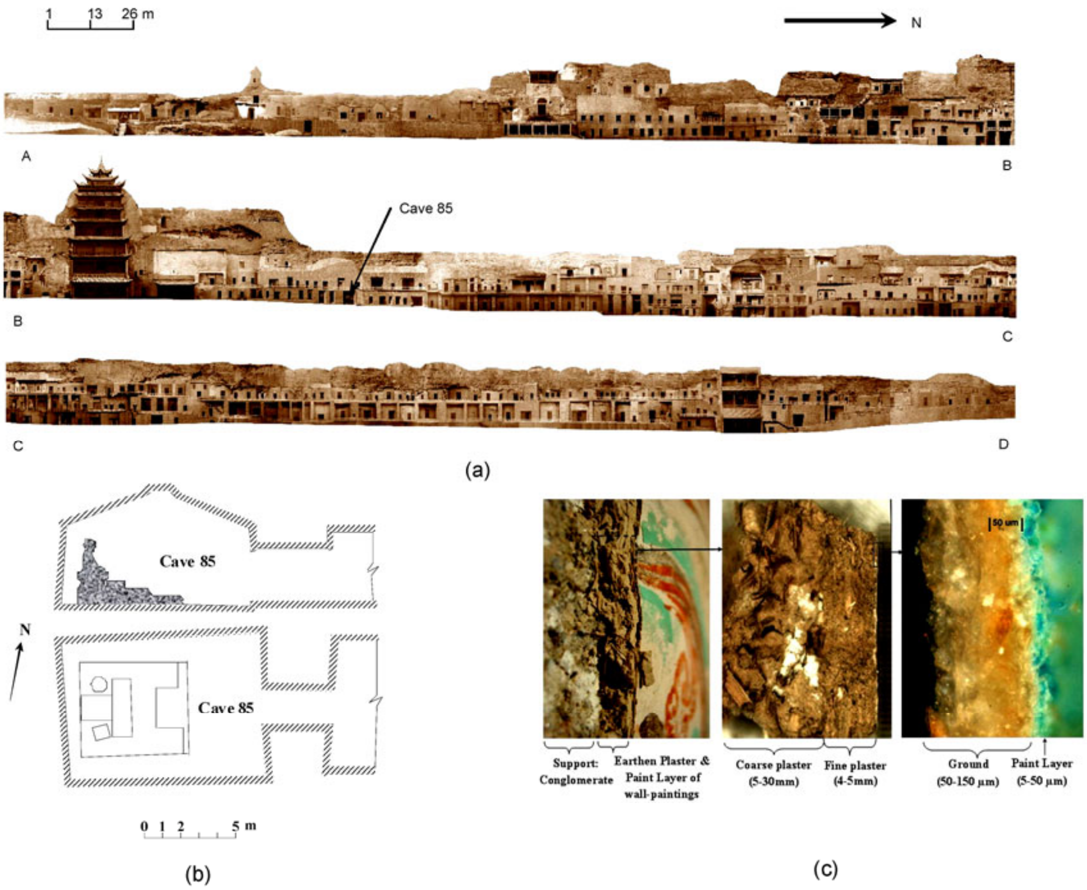


Fig. 1 Elevation drawing (a), representation cave section (b) (Piqué *et al*., 2010) and stratigraphy of the painted plaster (c) (Xu *et al*., 2010) at Mogao Grottoes

Many researches have dealt with the deterioration of porous materials due to salt weathering (Charola, 2000). With respect to salt weathering, it is found that, moisture transport within a pore system and its changes resulted from environmental variations, i.e., air temperature (AT) and RH, are very important to understand deterioration mechanisms and patterns (Rodriguez-Navarro and Doehne, 1999).

This paper aims to investigate moisture exchanges between the wall paintings and air in the caves by adsorption-desorption tests in the laboratory. This study also uses the unsaturated soil mechanics to elaborate on the water vapor balance in varying RH. Special attention is paid to the basic mechanism of moisture adsorption-desorption induced by humidity and temperature changes in wall paintings to understand the general transformation and distribution of moisture during the process of environmental changes. The research tries to provide a basis for controlling the microenvironment in caves to prevent salt disasters.

2 Real-time monitoring of air temperatures and relative humidity at the Mogao Grottoes

A systematic monitoring network, built by Dunhuang Academy in the last two decades, recording AT, RH, wind velocity, airborne dust, etc., aimed to investigate the relationship between wall painting deterioration and environmental conditions.

Cave 85 contains a wealth of art, rich information on religious practices and daily life and the entire painted surface is approximately 350 m² (Agnew and Li, 2010; Wang, 2010). The cave consists of an antechamber, corridor and main chamber with a large altar platform and sculpture group. Its main chamber has dimensions of approximately 10.70 m×9.45 m ×5.40 m (Fig. 1b), in which serious deterioration of wall paintings were afflicted by moisture migration (Piqué *et al.*, 2010; Xu *et al.*, 2010). Historically, river water once entered the cave, and it gave rise to the migration of capillary water along the mural, triggering off paint flaking and black spots on the lower part of the wall. Laboratory tests demonstrate that a stable climate in the cave below 67.00% RH is necessary for the best preservation of wall paintings (Agnew *et al.*, 2010).

The microclimates in Cave 85 have been measured since the 1990s to monitor the deterioration development. Fig. 3 shows the ATs and RH variances recorded in Cave 85 in 2006. It shows that AT change inside the cave was discovered to be less than the counterpart outside. However, the humidity inside the cave was likely to have been influenced by the humidity outside the cave, especially between May and October. Fig. 4 illustrates that AT and RH dramatically changed outside the cave on July 1, but the interior parameters remained almost the same on Feb. 1. During the winter, as shown in Fig. 5, sudden alterations of RH and AT outside the cave were evidently found at sunrise and sundown.

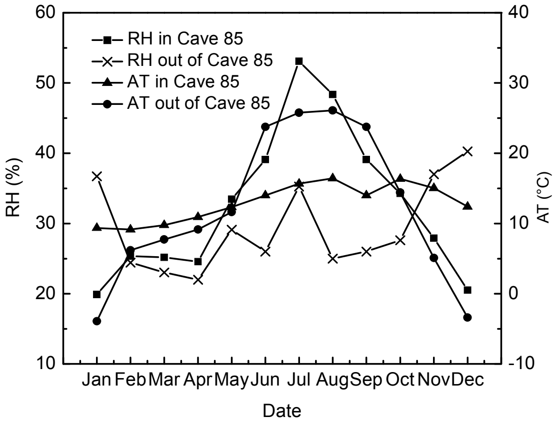


Fig. 3 Air temperatures and relative humidity monitored in and out of Cave 85 in 2006

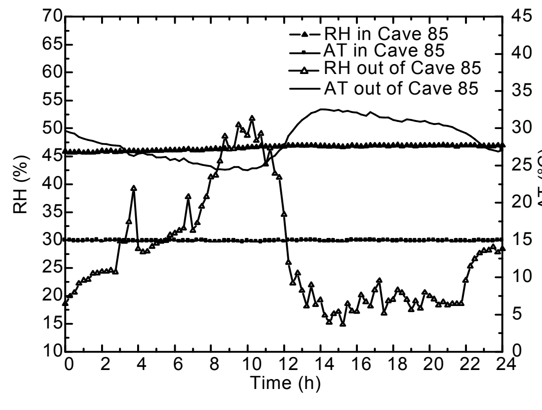


Fig. 4 Air temperatures and relative humidity monitored in and out of Cave 85 on July 1, 2006

The data above indicate that the interior environment in the cave keep relatively constant in a daily-scale, but varied in an annual-scale. It was the inclement weather that caused the microclimate in the cave to be changed.

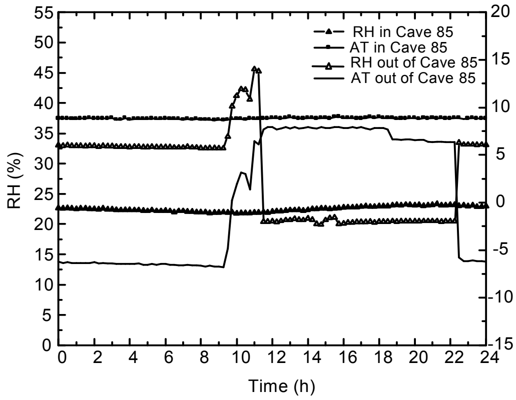


Fig. 5 Air temperatures and relative humidity monitored in and out of Cave 85 on Feb. 1, 2006

3 Methods and materials

3.1 Test instruments

3.1.1 Humidity control unit

Humidors as shown in Figs. 6 and 7 are employed to maintain a controlled humidity environment, in which supersaturated saline solutions, such as potassium sulfate (K_2SO_4) and lithium chloride (LiCl), were used. According to solution chemistry, it is known that a specific RH can be kept over an enclosed surface of the saturated salt solution (Butt *et al.*, 2006). For example, 23.11% RH and 97.59% RH can be achieved by LiCl and K_2SO_4 at 20 °C, respectively, as listed in Table 1.

3.1.2 Temperature control unit

The humidors are put into the plant growth cabinet (TPG-1260-5×400-TH, Australia) that is regarded as a temperature console cabinet for AT control (Fig. 8). Based on experimental requirements, the temperature can be synchronously monitored by the console cabinet.

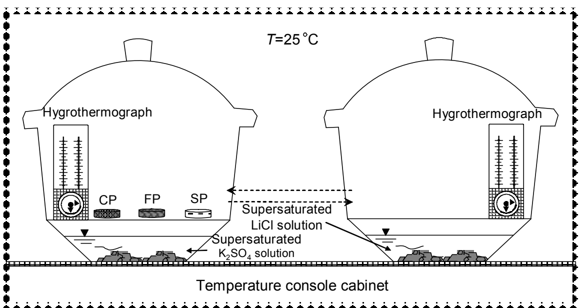


Fig. 6 Schemes of moisture adsorption-desorption tests under a constant temperature (CP: coarse plaster; FP: fine plaster; SP: silt plaster)

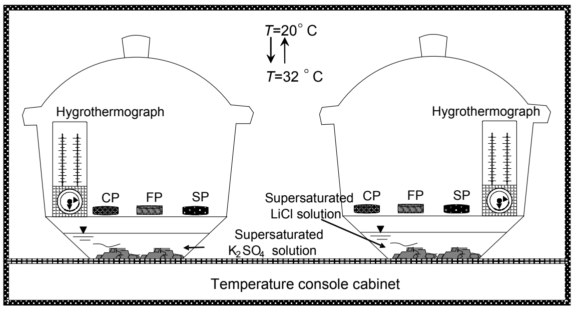


Fig. 7 Schemes of moisture adsorption-desorption tests under varying temperatures (CP: coarse plaster; FP: fine plaster; SP: silt plaster)

Table 1 Relative humidity controlled by saturated salt solutions (Butt *et al.*, 2006)

Saturated salt	RH (%)					
	10 °C	20 °C	25 °C*	30 °C	32 °C*	40 °C
LiCl solution	11.29	23.11	22.26	21.61	19.53	11.21
K_2SO_4 solution	98.18	97.59	97.29	97.00	96.88	96.41

* The relative humidities at 25 °C and 32 °C are linearly interpolated

Fig. 8 Plant growth cabinet

3.2 Materials

The typical structure of wall paintings (Fig. 1c) is composed of coarse plaster (CP), fine plaster (FP) and paint layer at the Mogao Grottoes. Wall paintings are made out of steps referring to daub the wall rock with coarse and FPs and then the grounding layer on the plaster layer for further painting when the caves were dug out.

To simulate the original composition of wall paintings as much as possible, three kinds of specimens were prepared (Table 2). Natural sediments (Fig. 9) taken from the riverbed of Daquan River, 180 m away from the grottoes cliff, were firstly sieved and then separated into two groups: sand (2–0.05 mm) and slit and clay (<0.05 mm), and then remixed together artificially: 34.95% sand (2–0.05 mm), 57.14% silt (0.05–0.005 mm) and 5.00% clay (<0.005 mm). The soil dust was mixed with 2.91% of plant fiber (such as wheat straw or hemp fiber) in a ratio of 100:3 in mass, and was followed by the addition of water up to the soil plastic limit. The mud was loaded into a mold to form a disk-like shape. The material compositions and specimen craftsmanship were obtained via the reverse analysis to wall paintings available (Zhao *et al.*, 2005). Fig. 10 shows the microstructure of the specimens.

3.3 Test procedures

Moisture adsorption-desorption tests were conducted on specimens to simulate the wall painting plaster that had experienced the humidified and dehumidified process in natural conditions (Fig. 11) by repeatedly changing specimens ambient RH or ATs with time cycle.

Two kinds of tests were designed to investigate the separate effects of ATs and RH. For the first design, the RH was controlled to vary between 22.26% and 97.29%, while kept AT constant at 25 °C (Fig. 11a). The second was planned with RH almost constant, for instance at approximately 20.26% and 97.29%, but AT varied from 20 to 32 °C (Fig. 11b). For easy understanding, the former was named as the "constant temperature test", while the latter as the "varying temperature test". Bui *et al.* (2009) carried out outdoor tests on rammed earth. However, their limitation lay in the environmental factors beyond accurate control.

3.3.1 Constant temperature test

In the condition of a constant temperature (Fig. 6), the designed AT was 25 °C and RH was 22.26% and 97.29%, controlled by supersaturated LiCl solution and supersaturated K_2SO_4 solution, respectively (Table 1). At first, three kinds of specimens (Table 2) were placed into the humidor with RH

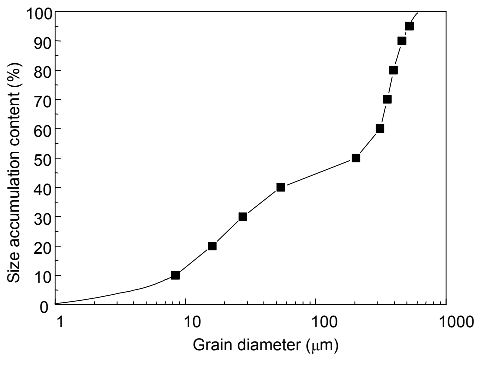


Fig. 9 Grain size distribution of natural sediments from Daquan Riverbed

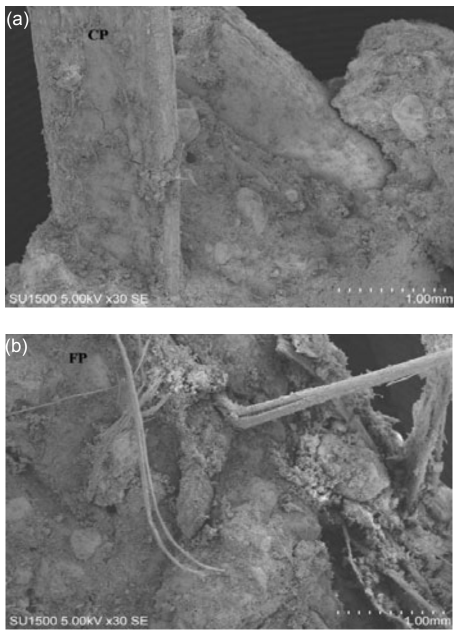
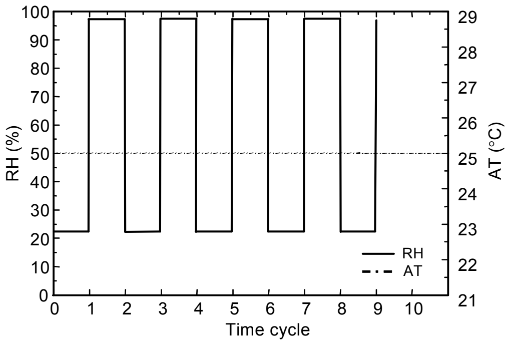


Fig. 10 SEM photos of the simulated coarse plaster (a) and fine plaster (b)

Table 2 Properties of the specimens

Specimens analogy	Size* (cm×cm)	Density (g/cm^3)	Proportion (%)	Original moisture (%)	Description
Coarse plaster (CP)	5×1	1.78	2.63	0.80	Sedimentary silt reinforced with wheat straw
Fine plaster (FP)	5×1	1.75	2.57	0.80	Sedimentary silt reinforced with hemp fiber
Silt plaster (SP)	5×1	1.66	2.71	0.80	Natural sedimentary silt as a control

* Diameter×height



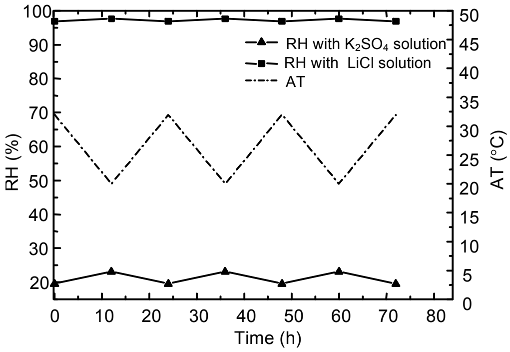


Fig. 11 Control process of moisture adsorption-desorption test in a constant temperature 25 °C (a) and varying temperatures ranged from 20 to 32 °C (b)

97.29% (supersaturated K_2SO_4 solution) for moisture adsorption until the specimen weight did not increase. Following the above procedures (Fig. 6), the specimens were then put into another humidor with supersaturated LiCl solution for moisture desorption. When there was no change in weight, specimens were then transported into the first humidor with supersaturated K_2SO_4 solution to continue moisture adsorption.

3.3.2 Varying temperature test

Under varying temperature condition (Fig. 7), specimens were either set into the humidor with supersaturated K_2SO_4 solution or LiCl solution for the test. Both humidors were put into the temperature console cabinet (Fig. 8) to operate the cyclic variations of temperature from 20 to 32 °C (Fig. 7). The RHs were calculated as 19.53% of LiCl and 96.88% of K_2SO_4 at 32 °C by the interpolation of the data, respectively (Table 1). Noticeably, the test followed after an AT was set at 32 °C in the cabinet till the weight unchanged. The AT and the weight of specimens were recorded in a 2-h interval.

4 Results and discussion

4.1 Weight changes in the constant temperature test

Fig. 12a shows the weight changes of CP specimens with varying humidity but constant temperature. It also shows that the weight of the specimens increased while being kept at a higher RH which was attributed to the specimens adsorbing water from humid air. In the case of the lower RH, the weight of the specimen decreased due to the specimens releasing water to dry air. It was of great interest to note the differences between the duration of adsorption and desorption. Further, it took about 40 h for CP to release water to a constant weight during a desorption process. Conversely, the adsorption process lasted for 60 h. Similar phenomena could be found on FP and SP as shown in Figs. 12b and 12c which underwent two moisture adsorption-desorption cycles in the constant temperature of 25 °C.

Fig. 12 shows the weight change of the specimens, clarifying that the specimens adsorbed air moisture when the air was humid but desorbed moisture in a dry air condition. Note that the adsorption duration was twice as long as that of desorption one, which meant wall painting plaster adsorbed moisture with more difficulty than released moisture. This indicates that closing all grottoes to visitors in the rainy summer is a good preventive measure against wall paintings deterioration. In addition, shortening the open time in summer could restrict the moisture adsorption and further, the adsorbed water might be released out during the close period.

4.2 Weight changes in the varying temperature test

As previously described in the varying temperature test, specimens were kept in either a high or low RH environment. In this study, specimen CP was firstly kept in the humidor with 97.59% RH at 32 °C until the specimen weight unchanged. Then, the temperatures from 20 to 32 °C were operated for cyclic variations. Fig. 13a shows the weight changes of specimen CP followed with temperature cycling. Note that the specimen weight gradually increased with temperature cycling, which implied an accumulated moisture adsorption of specimen.

With respect to the specimen CP, Fig. 13b shows the weight changes in the case of the same temperature condition as the previous one but in a low RH. As opposed to Fig. 13a, Fig. 13b illustrates an accumulated moisture desorption from the specimen.

The accumulated moisture adsorption was also reported by FP (Fig. 14a) and SP (Fig. 15a) when high humidity condition existed. Similarly to Fig. 13b, an accumulated moisture desorption was reflected on FP (Fig. 14b) and SP (Fig. 15b).

The test data as shown in Figs. 13–15 indicate that it was the humidity rather than the type of specimen that determined the final "wetting" or "drying" trend of the specimen under a varying temperature condition.

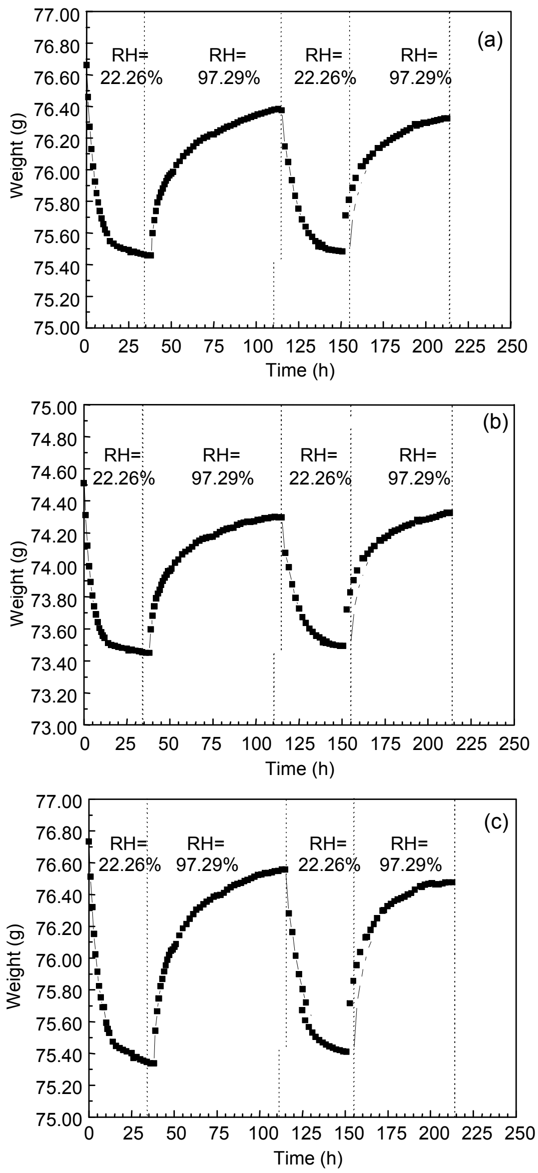


Fig. 12 Weight changes of (a) coarse plaster, (b) fine plaster and (c) silt plaster at 25 °C during high and low RHs

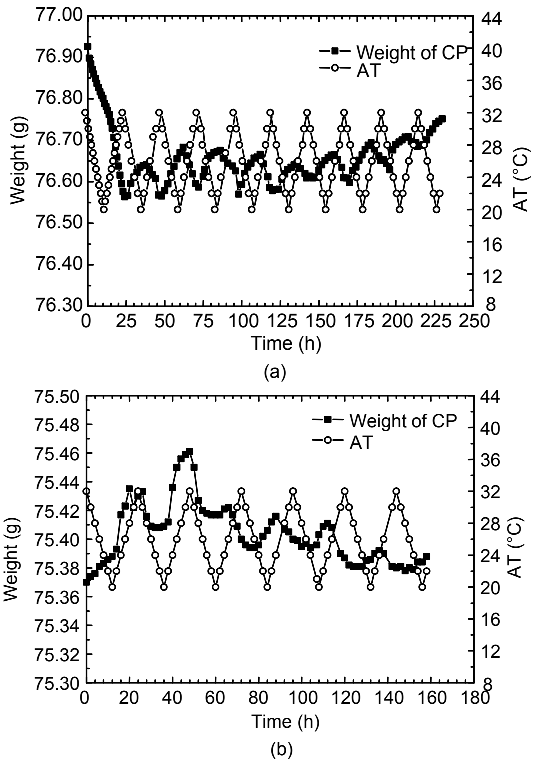


Fig. 13 Weight changes of coarse plaster at the temperature cycle from 20 to 32 °C during (a) high RH and (b) low RH

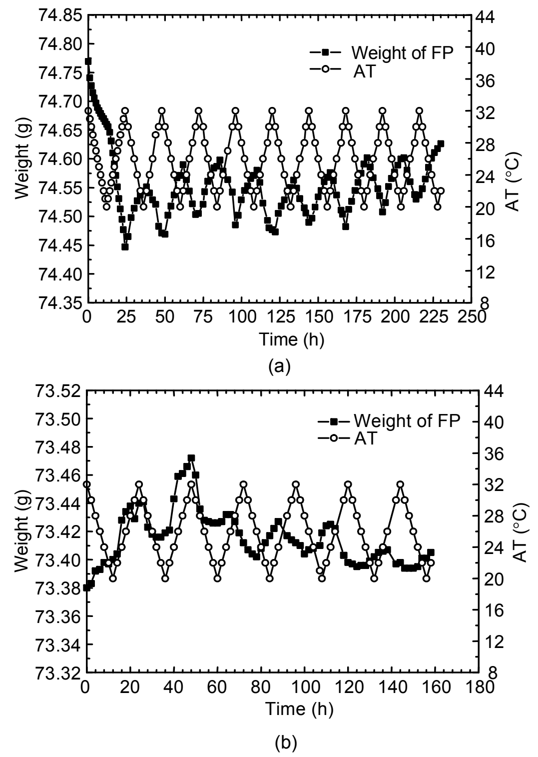


Fig. 14 Weight changes of fine plaster at the temperature cycle from 20 to 32 °C during (a) high RH and (b) low RH

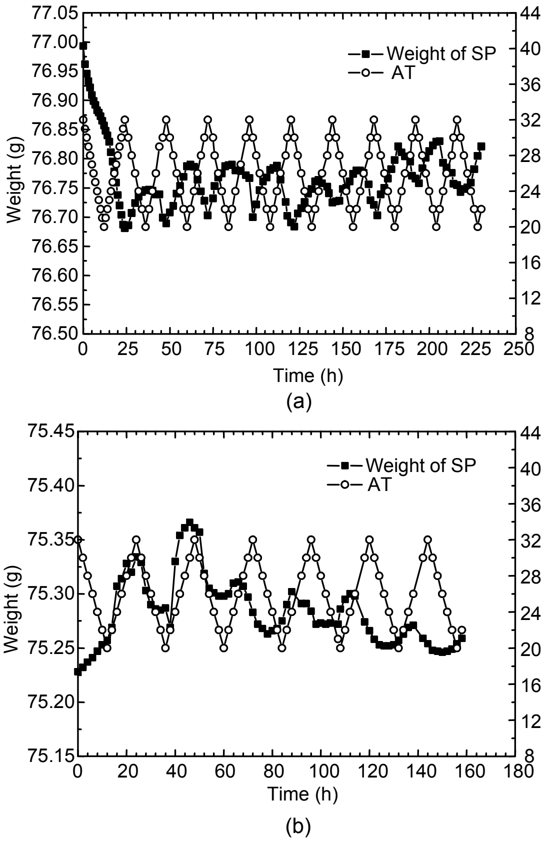


Fig. 15 Weight changes of slit plaster at the temperature cycle from 20 to 32 °C during (a) high RH and (b) low RH

4.3 Moisture condensation in porous media

The complex adsorption-desorption process depends on the chemico-physical characteristics of both the atmosphere and the surface of the porous media. The RH within a pore is a function of temperature, mixing ratio, pore geometry, and presence and nature of soluble salts, and is different from pore to pore (Fredlund and Rahardjo, 1993; Camuffo, 1998). This research found that the porosity of the wall paintings in the Mogao Grottoes changes seriously, especially in the plaster layer (Fig. 1c).

With respect to the condensation process in porous media, Camuffo (1988) introduced two basic types of pores (open and internal pores) to illustrate the situation. Open pores, like a hemisphere, or a portion of a hemisphere with very large outlets compared to the pore volume (Fig. 16a), are found on the surface of soil bodies. In the soil porosity, for each open pore, condensation begins at a low critical RH(r_p) determined by the effective radius of curvature of the pore r_p. The smaller the pore, the lower the RH required for equilibrium with the water meniscus. The ambient RH on the meniscus is related to a warped surface. When the RH increases, condensation occurs and the radius of curvature of the meniscus (r_m) increases, and vice versa. As a consequence, the process is irreversible.

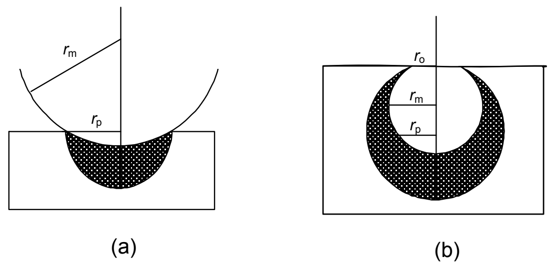


Fig. 16 Condensation in an open pore (a) and an internal pore (b) (Camuffo, 1998)

Internal pores, connected to the atmosphere by small outlets facing the surface or entering other pores or capillaries, are typically found inside soil bodies (Fig. 16b). On a certain meniscus, there must be an initial condensation value at the low critical RH=RH(r_p), which is in equilibrium with the radius of curvature of the pore r_p. During the adsorption-desorption process, there is an increasing condensation and a decreasing free space into the pore and so the radius of curvature of the new meniscus changes. On a certain meniscus, there must be an initial condensation value at the low critical RH=RH(r_p), which is in equilibrium with the radius of curvature of the pore r_p. During the adsorption-desorption process, there is an increasing condensation and a decreasing free space into the pore and so the radius of curvature of the new meniscus changes. The smaller new radius r_m needs new lower equilibrium RH(r_m).

However, inside the cavity, the actual RH was under a balanced state and the greater radius of curvature of the pore r_p, now corresponds to supersaturation for the smaller r_m. Thus, if RH(r_p)>RH(r_m), condensation is accelerated. Therefore, the initial level RH(r_p) of balance station cannot be obtained until complete filling of the pore and the process is irreversible.

Comparing condensation process of "open pores" with "internal pores", it is reasonable to predict that the condensation of internal pores is easier than for that open pores in the plaster layer of wall paintings.

During the desorption process inside the pore, evaporation is triggered when the ambient RH drops below a critical value. After some evaporation, the RH in equilibrium with the meniscus RH(r_m) becomes relatively high in comparison with the external RH which is RH<RH(r_o). Thus, different RH on both sides of the pores occurs, and the external condition favors further evaporation to keep balance of the RH. Consequently, the process is accelerated and is irreversible. Evaporation continues until all the liquid water inside the pore, except strongly bond water, has evaporated.

During the adsorption-desorption process of the plaster layer of wall paintings, the condensation -evaporation cycles, being thermodynamically irreversible, present noticeable hysteresis as varying AT and RH.

5 Implication of test data to conservation of wall paintings

More than 3×10^5 visitors a year have visited Mogao Grottoes since 2001. Statistics show that visitors are mainly concentrated in the period from July through September and peaked from 1:00 to 3:00 pm. Cave doors being open for visitation allows rapid infiltration of outside hot and humid air into the caves. An increased humidity of the interior air is considered to be harmful to the wall paintings, e.g., leading to the activation of salt disease.

This research tries to distinguish the separate effect of temperature and humidity from a viewpoint of adsorption-desorption. Test data reveal the importance of temperature variation on the moisture adsorption in addition to that of high humidity. High humidity, of course, is responsible for the moisture adsorption to the wall paintings, but the variation of temperature at the same time will increase the moisture adsorption. This provides another hint in understanding the disadvantageous effect of opening the entrance doors to visitors too often in summer. Frequent opening and closing of the entrance door for different visitor teams results in a heating-and-cooling effect of interior air due to the frequent infiltration of hot air outside the caves. This is harmful to the wall paintings in caves at ground level, where comparatively high humidity is produced because of capillary suction from ground. Demas *et al*. (2010) and Maekawa *et al*. (2010) indicated that salt mixtures from Cave 85 adsorb much moisture when the relative humidity increased more than 67.00%, leading to the activation of salt disease. Therefore, this research implies that frequent opening and closing of the entrance door of the caves, especially at ground level, would be better to avoid. It is suggested that visitor teams should be managed in close connection, one after another, and during the visitation period the door can be kept open instead of frequent opening and closing. Cutting the lag time between two visitor teams can reduce the total duration time of the doors being open, which helps restrict the adsorption and therefore, increase the recovery of wall paintings by extending the desorption period when caves are closed.

6 Conclusions

Separate effects of temperature and RH on moisture adsorption and desorption of wall paintings at the Mogao Grottoes in China were investigated by newly designed laboratory tests. The results indicate that high humidity is the first important factor that contributes to moisture adsorption. It is also found that temperature fluctuation tends to enhance an accumulated moisture adsorption at high RH or accumulated moisture desorption at low RH. It is suggested that the entrance door of the caves at ground level should be opened for visitation as short as possible and, during the visitation period, the door should be kept in a continuously opening instead of frequent opening and closing for prevention of wall paintings deterioration.

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