

Electrochemical Desalination Test of Bricks as a Building Material for Historical Buildings in Japan

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ABSTRACT: Brick buildings and structures are often exposed to outdoor condition, and deterioration of the bricks due to salt weathering caused by the surrounding environment has been reported in various parts of Japan. In Japan, not only the preservation of cultural properties but also their utilization is currently being promoted, and the beauty of brick surfaces is at a stage where it is becoming more important. For these reasons, a simple, low-cost, easily installed desalination model to desalinate only those areas where salt weathering was observed as first aid of deteriorated bricks was created. Powdered cellulose and copper plates were used as electrodes and these materials are readily available and easy to handle for professionals of conservation science as well as non-professionals. The aim of the research presented in this paper was twofold: to investigate the desalination effect of a simple electrochemical desalination model and to obtain knowledge for practical tests by conducting experiments under different energization conditions and observing the surface of the bricks after energization. Na_2SO_4 solution was used in the experiments and the brick samples containing Na_2SO_4 were used for desalination test by energizing for 8 days and sample exposure test after energization. When powdered cellulose and copper plates were used as electrodes, it was found that when sufficient water was supplied, approximately 64% of sulfate ions in the brick sample were removed when the energization conditions were 5 V and 0.5 A and 73% of sulfate ions were removed when the energization conditions were 5 V and 1 A. Visual observation confirmed that this removal rate suffices in preventing salt precipitation after energization is applied. This desalination method is expected to be suitable for Japanese historical bricks, which have varied characteristics, because it is possible to adjust the amount of water supplied during the energization by using an easily removable powdered cellulose for the electrode, and desalination can be performed without damaging the brick surface. However, it was found that the black areas consisting mainly of Cu_2O were formed after the 8-day energization. Since the efficiency of desalination from this area to the anode may be low, this remains a challenge for the future.

KEYWORDS: Brick, Conservation science, Desalination, Modernization heritage, Salt weathering

1. Introduction

In recent years, Japanese brick structures have been attracting attention as industrial and modernization heritage. Many brick buildings and structures were constructed throughout Japan as symbols of modernization. However, a great amount collapsed during the Great Kanto Earthquake of 1923 [1]. Since then, bricks have rarely been used as construction materials in Japan, which is prone to frequent earthquakes. For this

reason, the brick buildings and structures that were constructed before 1923 and remain in Japan are extremely valuable and have historical and artistic value.

However, these brick buildings and structures are often exposed to outdoor condition. In Japan, not only the preservation of cultural properties but also their utilization is currently being promoted, and the beauty of brick surfaces is at a stage where it is becoming more important. However, brick deterioration due to salt

weathering caused by the surrounding environment (Figure 1 and Figure 2) has been reported in various parts



Figure 1: Buildings showing signs of deterioration due to salt weathering



Figure 2: Bricks with peeling due to salt weathering

of Japan. Responding to this issue, the application of a hydrophilic resin was tested to prevent the penetration of water, which is one of the causes of salt weathering [2], [3]. Other attempts have been made to treat bricks with penetrating coatings, which have been reported to be effective in inhibiting salt weathering in Japan [4]. More recently, other methods have been used to adjust the temperature and relative humidity environment through numerical analysis and simulation to predict the occurrence of salt weathering from the perspective of environmental engineering [5], [6], constructions to break the origin of moisture that causes salt weathering [7] and restoring the deteriorated parts of bricks with restoration materials [7], [8]. However, these methods require extensive construction, large amounts of money and manpower although municipalities and organizations that own brick cultural properties have limited budgets for the conservation and restoration of cultural properties, and there are challenges such as a lack of professionals in conservation of brick cultural properties and the shortage of local staff in Japan. Moreover, treatment or desalination methods for salt weathering using hydrophilic resin or penetrating coatings have yet to be thoroughly established. Additionally, few efforts have been made to remove salt within the Japanese environment, and there is

a need to develop more effective desalination methods to control the deterioration of bricks.

In this study, electrochemical desalination (ED) which has been mainly used to desalinate concrete structures was applied to remove the salt contained in bricks. ED is a method for removing or reducing the number of anions, such as chloride (Cl^-) and sulfate (SO_4^{2-}) ions, from a material by transferring them through electrophoresis. This method has been applied to concrete structures in Japan since 1992, and its effectiveness has been reported since [9]. While these promising effects can be expected to occur in bricks, there are few examples of Japanese historical building bricks being desalinated. Outside of Japan, an electrokinetic desalination test on bricks has been active. Various types of salt were the target of desalination in the tests and it was reported that 99% of chlorides and nitrates could be removed from the bricks using ED [10], [11]. However, sulfate was more difficult to remove completely than other salts and the removal rate of sulfate was reported to be only 89% [12]. Indeed, ED is still seemingly in the testing stage and has not yet been implemented. Moreover, it is necessary to select a more manageable material as an electrode to perform ED locally. In the case of Japanese brick cultural properties, not all brick walls are deteriorated due to salt weathering, and there is often variation in the areas where salt weathering is observed. Therefore, we consider that the time and cost of conservation treatment are able to be reduced by desalinating only those areas where salt weathering is observed, rather than desalinating the entire wall surface. However, desalination of only the necessary areas has not been considered. Therefore, as a first aid, it is necessary to develop a method to desalinate only those parts of the bricks that need desalination, without modifying the bricks that are not deteriorated. This study proposes that a viable solution to the aforementioned issues surrounding brick cultural properties is to create a simple, low-cost, easily installed desalination model for individual bricks. In other words, to create a simple desalination method that specifically targets the parts that require desalination rather than the entire brick wall.

For the research presented in this paper, sulfate, which is difficult to remove, was targeted for the desalination tests. A simple, low-cost, easily installed desalination model for individual bricks was created using materials that are readily available and easy to handle for professionals of conservation science as well as non-professionals. The aim of the research was to investigate the desalination effect of the simple ED model and to obtain knowledge for practical tests by conducting experiments under different energization conditions and observing the surface of the bricks after energization.

2. Salt weathering in bricks

Salt weathering in bricks is a phenomenon that occurs when water that has penetrated the bricks and joints

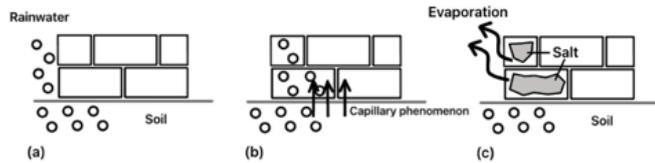


Figure 3: Schematic diagram of salt weathering process in bricks material

through groundwater or rain evaporates, causing the salts dissolved in the water to crystallize in the voids and on the surface, resulting in brick deterioration (Figure 3). When the stress exerted by the salts exceeds the tensile strength of the brick, brick failure occurs. [13]. Various types of salts, such as Na_2SO_4 (thenardite), $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$ (epsomite), and CaSO_4 (gypsum), have been detected or precipitated from deteriorated bricks. Additionally, it is known that different salts precipitate in different seasons and locations [14]. These salts progressively deteriorate the bricks through repeated precipitation and deliquescence. Na_2SO_4 (thenardite) is one of the most common salts that can deteriorate bricks, and is reported to be one of the most damage-inducing salts [15]. Furthermore, when the relative humidity changes from 70% to 100% at a temperature of 20°C, it undergoes a phase change to $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$ (mirabilite). Additionally, the hydration and dehydration process of these salts is more rapid than other salts and this phase change is repeated several times during the day and is accompanied by a large hydration pressure, which accelerates the fracture of the bricks. [16].

3. Methods

3.1. Brick samples

The bricks used as samples were made imitating the characteristics of the bricks used in the Tomioka Silk Mill, which were produced in Japan in the 1870s. Bricks made before 1887 were fired at a low temperature, resulting in a water absorption rate of about 17% and a compressive strength of about 9.5 N/mm², both of which are lower than modern brick standards. In addition, since the first Japanese brick standards were established in 1924, bricks manufactured before that date were considered to have varied characteristics [17]. The brick samples were each made to the following standards in order to make the brick samples large enough to be easily cut for evaluation after energization and to minimize waste after the experiments. Therefore, the brick samples were cut into 5 cm × 5 cm × 11 cm pieces for Test 1, and 5 cm × 5 cm × 4 cm for Test 2. Each brick sample was immersed in 35.5 g/L (0.25 M) Na_2SO_4 solution for 5 days, and dried until the moisture content was under 0.05%. The concentration of Na_2SO_4

solution was set at 0.25M, which was the concentration that did not affect the evaluation of the brick surface condition throughout the preliminary experiments. Table 1 shows the properties of the samples.

Table 1: Physical properties of brick samples

		Absolute dry weight [g]	Water absorption [%]	Volume [cm ³]	Dry density [g/cm ³]	Na_2SO_4 content [g]
Test 1	Control	592.45	17.91	275	2.15	5.14
	A1	556.29	18.44	275	2.02	3.19
	A2	621.96	18.8	275	2.26	4.86
	B1	581.75	18.87	275	2.12	4.03
	B2	589.41	18.65	275	2.14	5.55
	C1	586.42	18.81	275	2.13	5.70
Test 2	Control	147.79	18.11	100	1.48	1.64
	I	143.22	18.29	100	1.43	1.62
	II	161.33	18.22	100	1.61	2.03

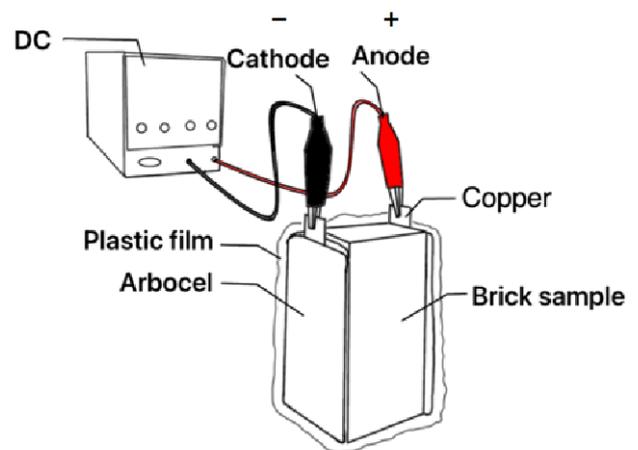


Figure 4: ED model (created by the author)

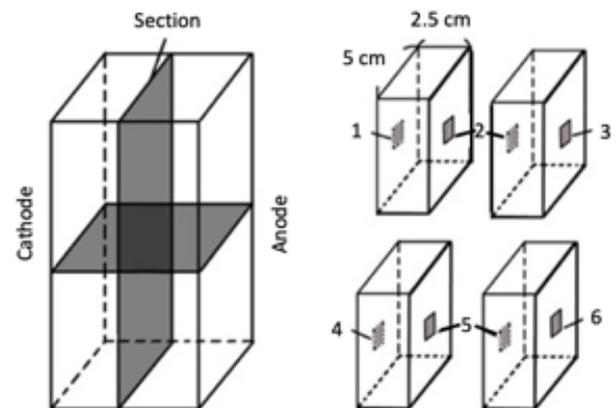


Figure 5: Schematic of the division of the sample (left) and the brick powder sampling point (right)

Na_2SO_4 solution was used in the tests. Na_2SO_4 (thenardite) is one of the most common and reportedly one of the most damage-inducing salts for bricks [15]. Furthermore, as mentioned above, it is more difficult to remove from bricks than other salts. For these reasons,

Na₂SO₄ solution was selected to be used in the experiments.

3.2. Process of the tests

3.2.1. Electrodes

Powdered cellulose (Arbocel BC1000 from Holland Gwabo) and copper plates (Kenis) were used to create the electrodes. Arbocel is originally used as an inert filler for cleaning rocks and frescoes and for conditioning poultices and has hydrophilic and insoluble properties [18, 19]. The copper plate (15 mm in width, 45 mm in length) had a purity of 99.96%. To 25g of dried powdered cellulose, ultrapure water with an electrical resistivity of 18.2 MΩ/cm was added, amounting to 20% of the dry weight of the brick samples. The mixture was combined until the Arbocel was moistened. Since the absorption rate of the brick samples was 20% of the dry weight, this amount of water was determined to represent the saturated water content and added to the powdered cellulose. Moistened Arbocel was placed on two sides and bottom of the brick sample, and copper plates were inserted into the lateral Arbocel layers, so that the copper plates did not touch the brick material. The test was conducted on the smaller surfaces of the brick (5 cm × 5 cm), namely the top and bottom, and the electrodes were placed at the top of the two sides. The surface was covered with a plastic film to prevent the Arbocel from drying out (Figure 4).

3.2.2. ED test under different energization conditions (Test 1)

In Test 1, the quantitative evaluation of the SO₄²⁻ concentration in the bricks was conducted by ED. Table 2 shows the voltage and current conditions applied to each sample. The energization period was 8 days. On the fourth day of energization, ultrapure water was added to the Arbocel of both electrodes in the amount of 20% of the dry weight. After the energization was completed, the samples were cut into half using a hammer and a blade, and further divided so that the top and bottom were equal. After cutting, approximately 3 g of brick powder was collected from the locations shown in Figure 5. After the test, ion chromatography and visual observation were used for evaluation. Visual observations focused on the brick surface conditions and on the form of salts that precipitated there after energization. The presence of any deterioration indications such as peeling or powdering on the surface of the bricks was also inspected.

3.2.3. Ion chromatography

Ion chromatography was used as the evaluation method for Test 1. The extract solution used for the analysis was prepared by grinding the collected brick

powder, then soaking it in ultrapure water, shaking it for 48 hours, and letting it stand for 24 hours. The volume of ultrapure water used was 1.5 ml, corresponding to 15 mg of brick powder. The extract was filtered through a 0.45

Table 2: Energization conditions of the samples used in the tests

		Voltage [V]	Current [A]	Additional water supply
Test 1	Control	0	0	×
	A1	5	0.1	○
	A2	5	0.1	○
	B1	5	0.5	○
	B2	5	0.5	○
Test 2	C1	5	1	○
	Control	0	0	×
	I	5	0.5	○
	II	5	0.5	×

○ Water was added on the fourth day of energization.

× Water was not added while energization.

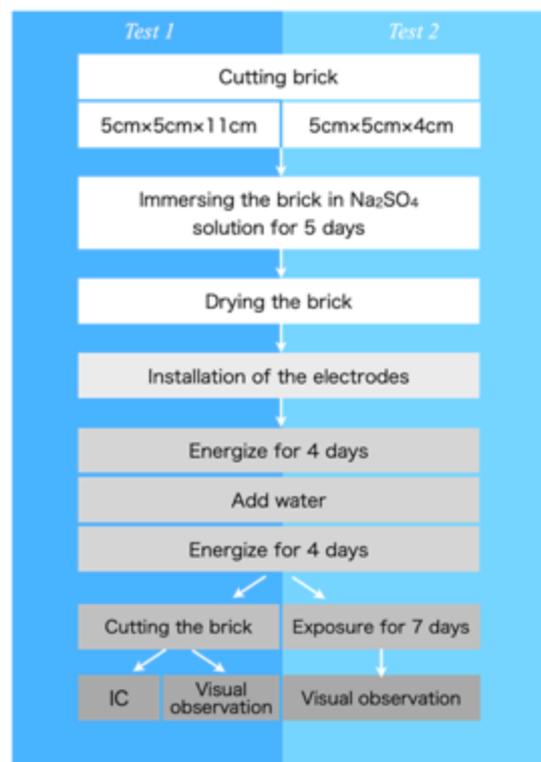


Figure 6: Flowchart of Test 1 and Test 2

μm diameter filter (Minisart RC4 from Sartorius) and then analyzed for anions. Metrohm 883 Basic IC plus was used as the analyzer. A separation column METRO A Supp 5 25/4.0 (6.1006.530) was used, and the flow rate was 0.70 mL/min.

3.2.4. Sample exposure test after ED test (Test 2)

Building on the results of Test 1, visual observations were conducted in Test 2 to evaluate the changes in the brick surface condition after energization. The samples

were exposed to an environment with a temperature of 25°C and a relative humidity of 60% for 7 days after being energized following the same procedures than those of Test 1. The same current-carrying conditions (5 V, 0.5 A) both with and without the addition of ultrapure water during energization were also prepared (Table 2). The flowcharts for Test 1 and Test 2 are shown in Figure 6.

3.2.5. Visual observation

Visual observation was conducted as an evaluation method for Test 1 and 2. For each test, the brick samples were dried after being energized and the presence or absence of salt precipitation was checked. In addition, for Test 1, the brick surface was observed using a KEYENCE Digital microscope VHX 900 in order to inspect the conditions of the brick surface in more detail.

4. Results

4.1. SO₄²⁻ concentration after ED test

The results of the ion chromatography analysis are described separately for the top and bottom of the brick samples. Regarding the top, the SO₄²⁻ concentration in the entire upper part of the brick samples decreased compared to that of the control sample when the current was applied at 0.1 A or higher. The decrease in SO₄²⁻ concentration in the middle and cathode side was especially pronounced when the energizing conditions were 0.5 A and 1 A. It was also found that under 0.5 A energizing conditions, SO₄²⁻ content in the brick powder collected from the top of the brick sample decreased by 35.5% compared to the control, and under 1 A, it decreased by 54.8%. However, as shown in Figure 7, SO₄²⁻ concentrations on the anode side were high, suggesting that SO₄²⁻ in the brick moved from the cathode to the anode, and some ions were removed from the brick by moving to the Arbocel on the anode side, while some remained on the anode side of the brick sample.

Regarding the lower part of the brick samples, there was no decrease in the SO₄²⁻ concentration in the entire lower part compared to the control brick sample, even when changing the current values. However, it was found that the SO₄²⁻ concentration on the cathode side decreased while it increased on the anode side when applying a current of 0.1 A or higher. Furthermore, when a current of 0.5 A or 1 A was applied, the SO₄²⁻ concentration decreased not only on the cathode side but also in the middle part (Figure 7). These results suggest that SO₄²⁻ in the lower part of the brick samples moved from the cathode to the anode when energization was applied under conditions of 5 V, 0.5 A or 1 A. However, SO₄²⁻ concentrations in the lower part of the brick samples did not decrease even when the energization was applied under 0.5 A or 1 A, which were

effective in desalination for the upper part of the brick samples, suggesting that SO₄²⁻ ions were not removed in the lower part of the brick samples and the desalination efficiency was lower than that of the upper part.

4.2. Presence or absence of salt precipitation

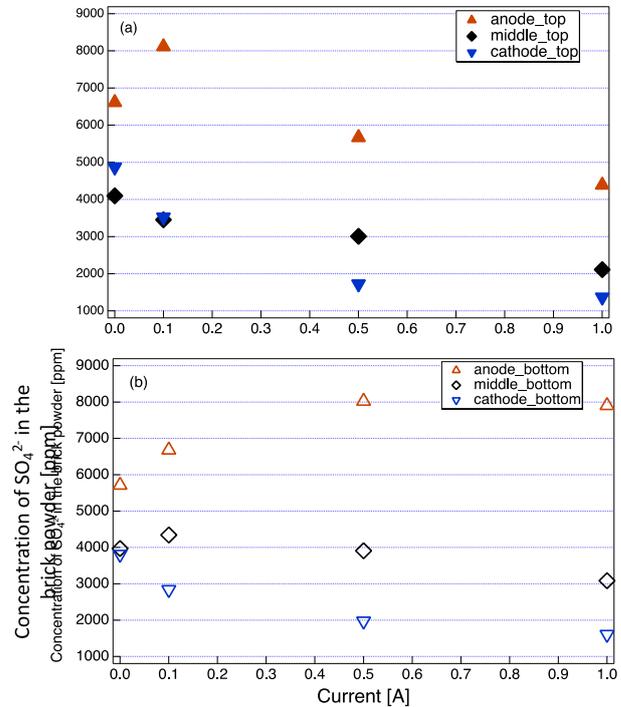


Figure 7: (a) Concentration of SO₄²⁻ in the upper part of the brick sample (b) Concentration of SO₄²⁻ in the lower part of the brick sample

Table 3: Conditions of the brick surfaces after Test 1

	Control	A1	B1	B2	C1
Whole picture					
Microphotograph					
Surface condition	<ul style="list-style-type: none"> Salt was deposited on the entire test piece. Peeling 	<ul style="list-style-type: none"> Salt was deposited on the entire test piece. Peeling 	<ul style="list-style-type: none"> No salt precipitation. 	<ul style="list-style-type: none"> Salt was slightly deposited on the entire test piece. 	<ul style="list-style-type: none"> Salt was slightly deposited on the entire test piece except the anode side.

Results of the Test 1 visual observations are shown in Table 3. The squares in the photos of the whole brick pictures (first row) indicate the areas where the sample surfaces were observed using an electron microscope. First, salt precipitation was observed on the entire brick sample when no current was applied. Additionally, the sample surface presented thin peeling due to salt precipitation. Furthermore, microscopic observation revealed that the salt formed needles and that brick grains were attached around the salt. Although the amount of salt precipitated in A1 decreased compared to that of the control sample, salt precipitated on the entire surface, and the microscopic images show that the brick surface

deteriorated through salt precipitation. On the other hand, only a small amount of salt precipitation or no precipitation at all were observed in B1, B2, and C1 where the current-carrying conditions were 0.5 A or higher.

Table 4: Conditions of the brick surfaces after Test 2

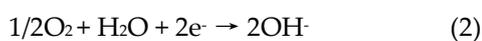
	Immediately after energization	Day 3	Day 7
Control			
	<ul style="list-style-type: none"> Salt in the form of needles precipitated all over the sample. Peeling, collapse 		
	<ul style="list-style-type: none"> Salt was generated on the surface from the black area to the anode side. The surface condition was good. 		
	<ul style="list-style-type: none"> Salt in the form of needles precipitated all over the sample. However, it was less than the control. 		

Microscopic observation revealed that the small amount of salt precipitated formed a film. The brick surfaces of these samples were in good condition, and no deterioration was observed in the form of peeling or powdering. The conclusion can be drawn from the visual observation results that the bricks are highly effective in desalination when energized under 5 V, 0.5 A conditions or higher. However, while the desalination effect was observed, it was found that black areas formed in these bricks after the energization.

The results of Test 2 are shown in Table 4. For Sample I, where water was added during the test, a black area formed (Figure 8), and salt was generated on the surface from the black area to the anode side. However, salt did not precipitate from the cathode side to the black area, and surface conditions were good. For Sample II, where no water was added during the test, no black area formed. Furthermore, salt precipitation was observed throughout the brick sample, although it was reduced.

5. Discussion

The reactions that have occurred at each electrode due to energization are shown below (1), (2).



From the results of Tests 1 and 2, it was found that the desalination of the bricks was made possible by energizing under the conditions of 5 V and 0.5 A or 1 A and the addition of water during the test, to the extent that

only a small amount of salt or no salt was deposited after energization. X-ray diffraction analysis of the salt precipitated after energization revealed $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ (thermonatrite) in addition to Na_2SO_4 . This is thought to indicate that SO_4^{2-} , which could not be removed from the

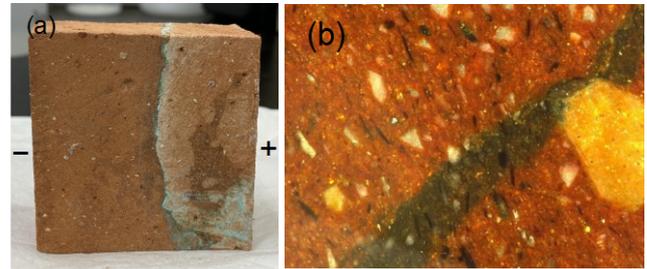


Figure 8: (a) The brick sample with the observed black area. (b) Enlarged photo of the black area.

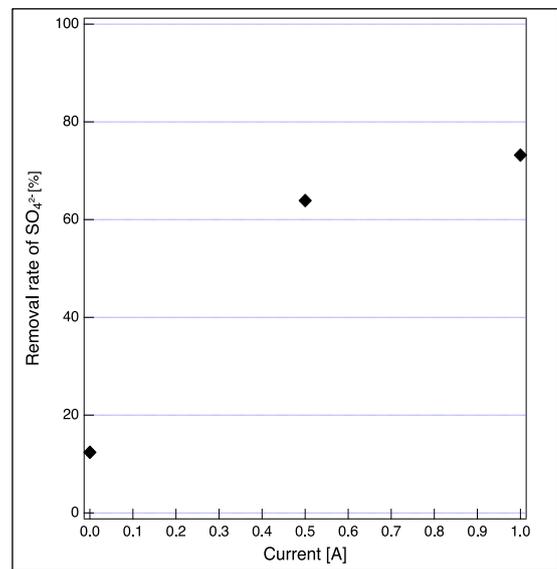


Figure 9: Change in removal rate of SO_4^{2-}

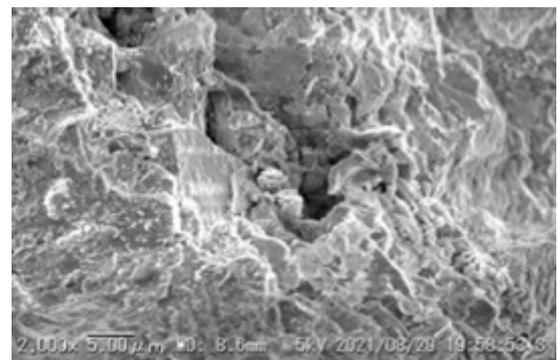


Figure 10: SEM image of the black area

brick by ED, precipitated as Na_2SO_4 , and that $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$ was formed by Na^+ remaining in the brick due to the removal of some of the SO_4^{2-} . This can be attributed to the fact that the SO_4^{2-} in the brick sample moved to the anode side during the energization and was fixed as $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ on the copper plate of the anode, resulting in a decrease in SO_4^{2-} concentrations. Since $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ was generated on the electrode as a product

of ED, the removal rate of SO_4^{2-} concentration in the brick sample was calculated using the weight change of the electrodes before and after energizing and the molecular weight of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$. Results indicate that when sufficient water was supplied to the brick sample, approximately 64% of SO_4^{2-} in the brick sample was removed when the energization conditions were 5 V and 0.5 A. Moreover, 73% of SO_4^{2-} ions were removed when the energization conditions were 5 V, 1 A (Figure 9). When a current of 0.5 A was applied, the removal rate of SO_4^{2-} increased by approximately 5 times compared to the removal rate when there was no current. However, when the current increased to 1 A, which is twice as large as 0.5 A, the removal rate dropped to about 1.1 times, and no significant differences were observed. Consequently, it is possible that the removal rate increases significantly when the current is up to 0.5 A, but that the relationship between the removal rate and the current value is low when the current is higher than 0.5 A. Since the number of samples tested was small in this iteration, it is necessary to increase the number of tests in the future and discuss subsequently. Although these removal rates are lower than the results of previous studies, visual observations showed that salt precipitation and deterioration of the brick surface could be reduced even after a current was applied, suggesting that ED current conditions are effective in inhibiting deterioration caused by salt weathering. At the same time, this ED model is expected to be suitable for Japanese historical bricks, which have varied characteristics, because it is possible to adjust the amount of water supplied during the energization by using an easily removable powdered cellulose for the electrode, and desalination can be performed without damaging the brick surface. In addition, the continuous fixation of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ on the copper plate of the anode caused even the Arbocel installed on the electrode to turn blue, and the migration of SO_4^{2-} to the electrode during energization could be visually confirmed. This is one of the advantages of using copper plates as electrodes. However, under energizing conditions where the desalination effect was observed, the efficiency of desalination at the lower part of the brick sample was low, suggesting that the length of the electrode was insufficient for the brick sample. Moreover, even though the energizing conditions for B1 and B2 were the same, visual observation after energizing showed a slight precipitation of salt in B2. This indicates that the reproducibility of the test may be insufficient.

Visual observation of Tests 1 and 2 showed a black area forming in the brick samples after energization. The black area was observed using scanning electron microscopy (SEM). Objects that appear particles were observed, as shown in Figure 10. Moreover, Cu_2O (Cuprite) was detected with X-ray diffraction analysis of

this area. According to Kawanishi, Cu_2O is formed in neutral aqueous solutions near room temperatures [20]. The pH of the area where the black part was formed was found to be 6.2, which is close to neutral. Additionally, it has been pointed out that metal elution occurs at the metal surface during the electrochemical process [21]. Therefore, it is possible that Cu^+ ions leached from the copper plate used as the electrode during the ED test and flowed into the brick sample.

For the reasons described above, it is possible that reaction (3) occurred. In other words, the black area, which was mainly composed of Cu_2O , may have formed in the neutral region in the process of copper ions eluting from the copper plate used as the electrode by energizing, flowing into the brick sample, and then moving to the cathode side.



In addition, since the desalination efficiency was low from the black area to the anode side, it is necessary to review the conditions to prevent the formation of black areas or to improve the desalination efficiency, even if black areas are formed.

6. Conclusion

The main conclusions derived from this study are as follows.

- When powdered cellulose and copper plates, which are materials that are easy to obtain and handle, were used as electrodes, it was found that when sufficient water was supplied to the brick sample, approximately 64% of SO_4^{2-} ions in the brick sample were removed if the energization conditions were 5 V and 0.5 A and 73% of SO_4^{2-} was removed by energizing under the condition of 5 V, 1 A conditions. Visual observation confirmed that this removal rate is sufficient to prevent salt precipitation after energization. At the same time, this desalination method is expected to be suitable for Japanese historical bricks, which have varied characteristics, because it is possible to adjust the amount of water supplied during the energization by using an easily removable powdered cellulose for the electrode, and desalination can be performed without damaging the brick surface. However, the desalination efficiency of the lower part of the sample was lower than that of the upper part, so the length of the electrode needs to be reconsidered.
- By using copper plates as electrodes, the formation of copper sulfate on the anode and the change in color during energization could be observed. This would be to the advantage of being able to visually confirming the desalination effect during energization that they

are desalinated when this model is installed by non-professionals on site.

- On the other hand, it was found that the black parts consisting mainly of Cu_2O were formed after the 8-day energization. Since the efficiency of desalination from this film to the anode may be low, it is necessary to change the material of the electrode to prevent the formation of black areas, or to study the conditions to increase the efficiency of desalination even if black areas are formed.

In addition, at present, the reproducibility of the current-carrying conditions that are effective in desalination is insufficient, so it is necessary to increase the number of tests under the same conditions to improve the reproducibility. The remaining challenges and future prospects are to develop this method into a practical model that can be applied to actual wall installations and to discuss sustainable electricity supply methods.

The future implementation of this ED model is expected to reduce the progression of brick deterioration and it will help small municipalities and organizations that are trying to preserve and manage brick cultural properties under the situation of lack of personnel and limited budgets because intensive desalination of only those areas where salt weathering is observed will reduce the time and cost of conservation treatment.

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