

Effect of immersion in NaCl solution on the electrical conductivity and the reduction of the shear bond strength of resin-modified glass-ionomer-cements after current application

Yuta MATSUKI¹, Hiroko SATO¹, Noboru KAJIMOTO², Emi UYAMA³, Shinya HORIUCHI¹, Kazumitsu SEKINE³, Eiji TANAKA¹ and Kenichi HAMADA³

¹ Department of Orthodontics and Dentofacial Orthopedics, Tokushima University Graduate School of Biomedical Sciences, 3-18-15 Kuramoto, Tokushima 770-8504, Japan

² Section of Bioengineering, Department of Dental Engineering, Fukuoka Dental College, Fukuoka 814-0193, Japan

³ Department of Biomaterials and Bioengineering, Tokushima University Graduate School of Biomedical Sciences, 3-18-15 Kuramoto, Tokushima 770-8504, Japan

Corresponding author, Kenichi HAMADA; E-mail: hamada.dent@tokushima-u.ac.jp

Advancements in dental cements have considerably improved their bond strengths. However, high bond strength often makes the removal of restorations difficult. Thus, smart dental cements that show controllable bond strength are required. A conventional resin-modified glass-ionomer-cement demonstrated a significant reduction in the bond strength after current application. However, for this system, the ions in the cement are released into the oral cavity, resulting in a reduction of the electrical conductivity and in losses of the expected on-demand debonding property. Herein, the effects of immersion in 0.9 and 15% NaCl solutions on the electrical conductivity and debonding properties were investigated. The cement immersed in 0.9% NaCl solution from 1 to 28 days maintained similar bond strength reductions after current application, whereas that in 15% NaCl solution initially showed no bond strength reduction after 1 day but exhibited an increase in the bond strength reduction after immersion for 28 days.

Keywords: Smart dental cement, Debonding-on-demand, Electrical shear bond strength reduction

INTRODUCTION

Advanced dental cement products today demonstrate excellent properties compared to those of the conventional ones¹⁻⁹. In particular, the higher bond strength provides many patients with more durable dental devices. However, this potentially causes difficulties in the debonding of some types of dental devices, such as in the removal of the abutment of cement-retained dental implants for periodic maintenance^{10,11}. Thus, a smart dental cement exhibiting controllable bond strength, *i.e.*, a high strength during usage and a considerably reduced strength for maintenance, is required.

In our previous work, a prototype resin-modified glass-ionomer-cement (RMGIC) containing an ionic liquid (IL) was developed to produce a smart dental cement¹². Results showed that the incorporation of IL to RMGIC imparted substantial electrical conductivity to the prototype cement. Additionally, the bond strength of the prototype cement was reduced in response to an electrical current application (CA). Therefore, the concept of a “dental cement debonding on-demand” was validated. In the following evaluations conducted, the prototype cement immersed in distilled water potentially lost its electrical conductivity and its debonding on-demand property with increasing immersion time¹³. The reduction in electrical conductivity was due to the elution of IL to distilled water. Furthermore, the RMGIC sample that did not have IL acquired electrical conductivity and exhibited a bond strength reduction in response to CA after immersion in distilled water. These

findings suggest that the electrical conductivity of the prototype cement could increase or remain constant with immersion in an ionic solution, such as saliva, different kinds of liquid seasonings, and drinks in an oral cavity.

In this study, an RMGIC without IL was stored in distilled water and sodium chloride (NaCl) solutions with two different concentrations. After immersion, the electrical conductivity and shear bond strength with and without CA were evaluated, and the effects of the concentration of NaCl solution, immersion period, and CA were estimated. In addition, fracture surfaces were analyzed to study the fracture mode and interface.

MATERIALS AND METHODS

The details of the experimental procedures are also described in previous papers^{12,13}.

Specimen preparation for the evaluation of the shear bond strength

A commercial product (RelyX™ Luting Plus, 3M Japan, Tokyo, Japan) was adopted as an RMGIC for evaluation, and Ti was used as an adherent to simulate the bonding between the abutment and fixture of conventional cement-retained dental implants. Two Ti rods (Ø 8 mm×10 mm and Ø 20 mm×10 mm) were used to assemble the specimens. The cross section of the rods was sanded using 600 grit SiC waterproof paper. Then, the circular bonding areas on the cross section of both rods were roughened by sandblasting at 0.4 MPa using Al₂O₃ particles with an average diameter of 50 µm. After

the uniformity of the surface was verified visually, the rods were rinsed ultrasonically in distilled water for 5 min. The cement was then spread on the cross section of the \varnothing 8 mm Ti rod, and it was set at the center of the \varnothing 20 mm Ti rod. The pair of rods was pressed at a pressure of 20 g mm⁻² for 5 min, and the excess cement was removed manually. The specimens were left either in distilled water or NaCl solutions (NaCl: 99.0%, Kanto Kagaku, Tokyo, Japan, concentration: 9 and 150 g/L, referred hereafter as 0.9 and 15%, respectively) after bonding for 30 min. Thereafter, specimens were immersed for 1, 7, 14, or 28 days, whereas some were kept in distilled water and 15% NaCl solution for 0.5, 1, or 3 h. Distilled water and NaCl solutions (100 mL for one sample, 37°C) were not exchanged. Specimens were also prepared without immersion in any liquid and were tested 1 day after cement mixing to evaluate the effect of immersion in liquids on the properties of the prototype cement.

Current application to the specimens

Using a programmable power supply (Type 7651, Yokogawa Electric, Tokyo, Japan), an applied potential of 19 V was applied for 30 s on the specimens. The \varnothing 20 mm Ti rod was used as the anode. The current response over time were recorded using a digital multimeter (Type TY720, Yokogawa Electric), and the total charge density (Cd) applied to the specimens was calculated from the time-integrated current values.

Bond strength evaluation

To evaluate the shear bond strength (σ_s), a universal testing machine (AGS-500A, Shimadzu, Kyoto, Japan) was used. The specimen setting in the compression test jig is schematically shown in our previous research¹³⁾. The crosshead speed was set to 0.5 mm min⁻¹. σ_s was calculated as follows:

$$\sigma_s = L/S \quad (1)$$

where L is the load at fracture, and S is the bonding area (50.24 mm² in this study). The specimens obtained with and without CA were tested to investigate the effect of CA on σ_s . Each group contained more than 14 samples.

Fracture surface observation and analysis

After σ_s evaluation, the fracture surfaces of the specimens were observed and photographed using a digital camera with an automatic white balance and exposure settings. Each image was printed on a paper, and the areas of cement residue image and Ti surface image were cut off manually. The total paper weight of cement residue areas and Ti surface areas was measured, and the area fraction of cement residue was calculated from the weight ratio.

Statistical evaluation

Statistical analyses on the Cd and σ_s data were done using Steel-Dwass test performed by the EZR software¹⁴⁾ (Saitama Medical Center, Jichi Medical University,

Japan). Statistical significance was accepted at a confidence level of 0.05. The multiple regression analysis was also done using the same software.

RESULTS

Charge density

Figure 1A shows the effect of the immersion period on the Cd of the specimens obtained with CA. On the other hand, Fig. 1B depicts the difference of the Cd values relative to those obtained from the samples immersed in distilled water for the same period. The results of the statistical analysis on the data in these figures are summarized in Table 1. The average Cd value of the specimens increased significantly until an immersion of 1 h in distilled water and decreased gradually thereafter. The value obtained after 28 days of immersion indicated the saturation of the reduction of Cd . At this duration,

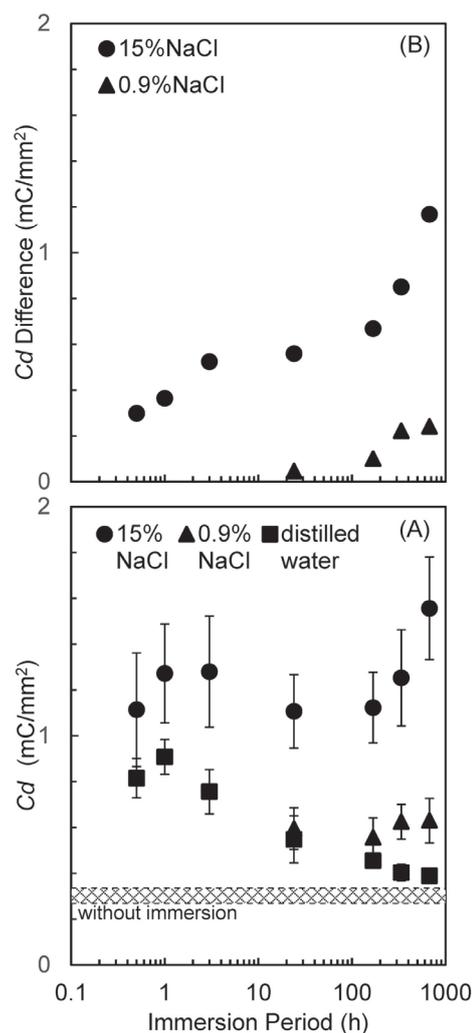


Fig. 1 Effect of immersion period in distilled water or NaCl solutions on (A) the charge density (Cd) values with CA and (B) the Cd difference from that immersed in distilled water.

Table 1 Statistical analysis results of the effect of immersion liquid or immersion period on charge density

Immersion liquid	Immersion period (h)						
	0.5	1	3	24 (1 day)	168 (7 days)	336 (14 days)	672 (28 days)
Distilled water	1	1	1	2, A	2, B	3	3
0.9% NaCl solution	/	/	/	4, A	4, B	4	4
15% NaCl solution	5	5, 6	5, 6	5	5	5, 6	6

Set of the same number and the same Roman alphabet indicates that there is no significant difference in the effect of immersion period, and that of the immersion liquid, respectively. Charge density of the specimen that were not immersed was significantly different from that of all the other immersed period.

the Cd calculated after immersion was only slightly higher than that obtained without immersion. For the specimens immersed in 0.9% NaCl solution for 1 day, the Cd value was significantly higher than that of the specimens without immersion but was not significantly different from that of specimens immersed in distilled water for the same time period. Beyond 1 day, the Cd value for specimens indicated a slight increase. Moreover, the Cd of the samples immersed for more than 14 days were significantly higher than those of the specimens immersed in distilled water for the same period. Using 15% NaCl solution, the Cd value increased until 3 h of immersion, and then decreased with immersion time up to 1 day. This was followed by a saturation in the reduction at 7 days immersion, and an increase from 7 to 28 days. However, only the Cd values of the samples obtained after 28 days of immersion showed a significant difference relative to those of the samples under different immersion periods in 15% NaCl solution. In general, the corresponding Cd values of the specimens immersed in 15% NaCl solution at different durations were significantly higher than those of the specimens immersed in distilled water and 0.9% NaCl solution. Additionally, the difference between the Cd of cement immersed in 15% NaCl solution and that immersed in distilled water increased monotonically with increasing immersion period.

Shear bond strength

Figures 2A, B, and C show the effect of the immersion time on σ_s . Table 2 shows the statistical analysis on these data. Figure 2D shows the reduction in the σ_s values of the specimen immersed in distilled water or NaCl solutions with CA. Without CA, all specimens showed similar σ_s with immersion time regardless of the immersion medium used. Although not necessarily significant, the reduction of σ_s values obtained from the samples without immersion to the samples immersed decreased until 14 days. Thereafter, σ_s value increased with increasing immersion time up to 28 days. In contrast, the σ_s reduction value with CA, as shown in Fig. 2D, was dependent on the immersion liquid used. The σ_s reduction values of the specimens immersed in distilled water decreased with increasing immersion time, whereas those of the samples immersed in 0.9% NaCl solution did not show a clear dependence on the

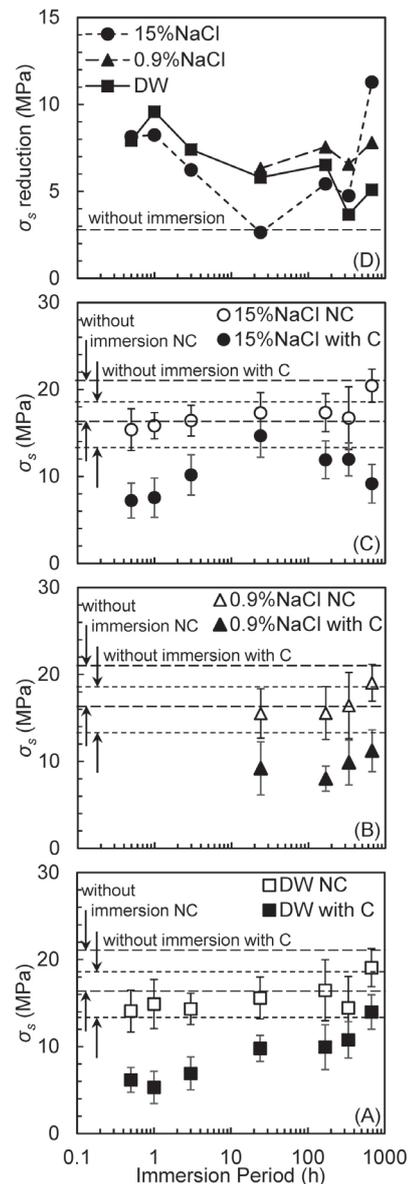


Fig. 2 Effect of immersion period on shear bond strength (σ_s) of specimen immersed in (A) distilled water, and (B) 0.9 and (C) 15% NaCl solution, respectively, and (D) that on σ_s reduction value with C. “NC” and “with C” means “without CA,” “with CA,” respectively.

Table 2 Statistical analysis results of the effect of immersion liquid, immersion period, or current application (CA) on shear bond strength

Immersion liquid/CA	Immersion period (h)														
	0	0.5	1	3	24 (1 day)	168 (7 days)	336 (14 days)	672 (28 days)							
NC	1	1	α	4	β	2	γ	δ	ε				3	ζ	
	2	3				5							4		
Distilled water	6	6	α	7	β	8	γ	9	δ	10	ε	6	11	ζ	
	7	11		13		16		11	A	12		15	12	D	
	8	12		14		17		13		14		16	15		
	9			15				18					17		
	10												18		
0.9% NaCl solution	NC	/						η	θ			κ	λ		
	with C	19	/						19	η	20	θ	21	κ	λ
		20	/							B	C				
15% NaCl solution	NC			22	μ	23	ξ	24			25	π			
													22	ρ	
													23		
													24		
													25		
with C	26	26	μ	27	ξ	28		30	A	31	π	32	29	ρ	
	27	30		33		36		33	B	34	C	35	37	D	
	28	31		34				36							
	29	32		35				37							

Set of the same number, the same Roman alphabet, and the same Greek alphabet indicates that there is a significant difference in the effect of immersion period, that of the immersion liquid, and that of the CA, respectively. “NC,” “with C,” and immersed period of “0 h” means “without CA,” “with CA,” and “without immersion,” respectively.

duration of the immersion. Finally, for the samples immersed in 15% NaCl solution, the σ_s reduction values decreased with immersion time until 1 day, and then increased until immersion for 28 days.

Effects of charge density on the shear bond strength

Figure 3 shows the effect of Cd on the σ_s of the specimen immersed in either distilled water or NaCl solutions. The σ_s values of the specimens immersed in distilled water and 0.9% NaCl solution decreased linearly with increasing Cd from 0 to approximately 1 mCmm⁻². In contrast, immersion in a 15% NaCl solution did not clearly indicate a linear dependence of the σ_s on Cd. Moreover, a larger Cd was required to reduce the σ_s of the specimens immersed in a 15% NaCl solution.

Fracture surface after the shear bond strength test

Figure 4 shows the typical fracture surfaces of the cathode of the specimens immersed in a 15% NaCl solution with CA after the shear bond test. Cement residues were hardly observed on the specimens produced after 3 h of immersion, indicating that the fracture was interfacial at the cathode. In contrast, the cathode surface was completely covered by the cement residues after 28 days of immersion, indicating that the fracture was interfacial at the anode, that

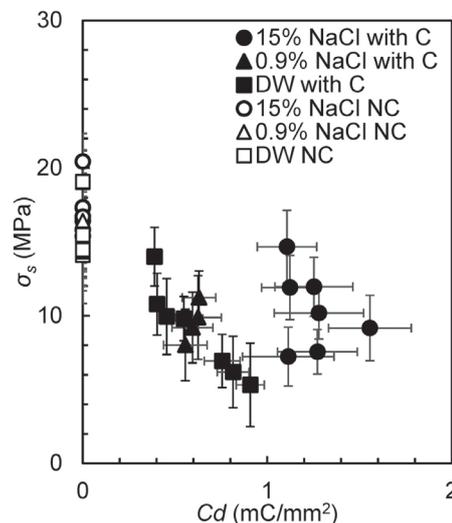


Fig. 3 Effect of Cd on the σ_s of the specimen immersed in distilled water, and 0.9 and 15% NaCl solution. “NC” and “with C” means “without CA,” “with CA,” respectively.

is, the fracture interface was inverted. The Ti surface became apparent and cement residues were found on

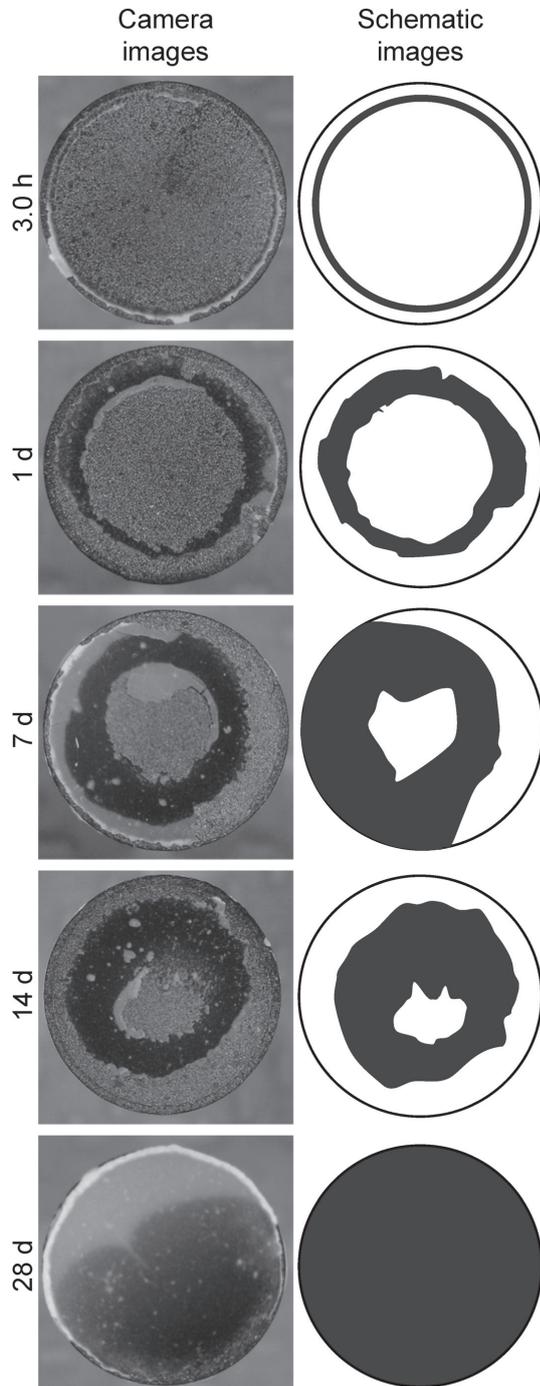


Fig. 4 Photographs of the fracture surfaces on the cathode of specimens immersed in 15% NaCl solution with CA after the shear bond strength test. Black area in the schematic images indicates the cement residue.

the fracture surfaces of the specimens immersed for 1, 7, and 14 days, indicating that the fracture occurred in a mixed mode. However, few specimens showed interfacial fracture. Figure 5 shows the effect of immersion period

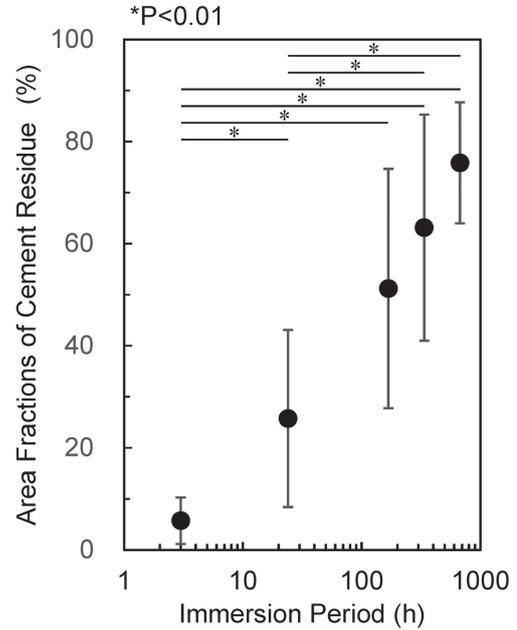


Fig. 5 Effect of immersion period on the area fraction of cement residue of the specimens immersed in 15% NaCl solution with CA after shear bond strength test.

on the area fraction of cement residues of specimens immersed in 15% NaCl solution with CA after the shear bond strength test. Although the difference was not necessarily significant, the area fraction was increased with increasing immersion period.

The fracture modes of the specimens immersed in distilled water and 0.9% NaCl solution exhibited a similar dependence on the immersion period. The specimen immersed in distilled water for 7 days or less with CA indicated interfacial fracture on the cathode, while those obtained after 14 and 28 days immersion indicated mixed-mode fracture. Interfacial and mixed-mode failure were also observed on the specimen immersed in 0.9% NaCl solution for 14 days or less, and for 28 days with CA, respectively.

Both the Ti surface and cement residues were apparent on the cathode fracture surfaces of the specimen without CA regardless of the immersion liquid used. This corresponds well with the results in our previous work¹³.

DISCUSSION

Fracture surface inversion

Without CA, the fracture mode and σ_s of the specimens in the present study were consistent with those of the specimens immersed in distilled water in our previous study. Moreover, the σ_s values obtained after CA were similar to those reported in our previous work. However, the fracture interface with CA observed herein was different from that in our previous research¹³. The

fracture with a shorter immersion period was interfacial at the anode in our previous research¹³⁾, while it was interfacial at the cathode in the present study. Although the specimen preparation procedures in both studies were similar, the interfacial fracture occurred at different electrodes. Interfacial fracture was possibly observed on both the anode and the cathode due to the occurrence of different electrochemical reactions such as the metal ion release on the anode and the hydrogen gas evolution on the cathode¹²⁾. Therefore, the transition without any change in the specimen preparation procedures may have caused some potential and slight differences. The hypothesis of this difference is as follows.

Although utmost effort was made to ensure that the specimen preparation procedure was consistent with the previously reported methodology, the manual mixing of the cement possibly resulted to differences in the properties, particularly the uniformity, of the cement mixture in the two works. As a result of the nonuniformity, the cement fluidity might have changed. It possibly affected the spread of cement, and thus, the thickness of the cement between the two Ti rods under the same pressure. The cement thickness affects the electrical resistance of the cement layer. Therefore, because the voltage applied to the specimen is constant, a change in the electrical resistance of the cement layer leads to a change in the voltage applied to the electrochemical reactions occurring at the anode and cathode interface. This change in the applied voltage possibly changed the type and/or the speed of the electrochemical reaction at the interface. The nonuniformity of the cement also possibly resulted to the nonuniformity of the electrical resistivity of the layer, which might also contribute to the deviations of the electrochemical reactions at the interface.

Another inversion of the mechanical failure mode was observed after the immersion of the samples in NaCl solutions with increasing immersion time as shown in Figs. 4 and 5. With increasing immersion duration, the concentration of NaCl that diffused into the cement increased. This led to the occurrence of an electrochemical reaction, the electrolysis of the NaCl solution. With this reaction, chlorine gas was generated on the anode, which damaged the interfacial bonding between the cement and the Ti surface. A concentric change from cathode to anode surface fracture should be observed because the NaCl concentration in the cement was higher near the perimeter than near the center due to NaCl diffusion from the perimeter facing the solution. However, as shown in Fig. 4, this clear concentric change was not observed. Instead, a mixed-mode fracture occurred. This mismatch was potentially caused by the nonuniformity of the cement mixture. However, in the area near the center of the cathode, cement residues were observed on the specimens immersed for 14 days or less, suggesting that the hypothesis was true.

Changes in charge density and shear bond strength with increasing immersion period

The increase in the Cd values of the specimens

immersed in distilled water for 1 h or less was caused by the diffusion of water into the cement, which enhanced the ion mobility in the cement layer. Subsequently, the release of ions to the water caused the observed decrease in the Cd values. These results suggest that the absorption and diffusion speed of water in the cement was higher than the diffusion and release rates of the ions in the cement.

The Cd values of the specimens immersed in 0.9% NaCl solution for 1 day were similar to those of the samples immersed in distilled water under the same duration. It slightly increased with increasing immersion period from 1 to 28 days but the change was not significant. However, the values for 14 and 28 days were significantly higher than those immersed in distilled water at the same immersion period. Therefore, immersion in 0.9% NaCl solution is effective in maintaining or increasing the Cd of cement, which leads to stabilizing or increasing the σ_s reduction value after a longer immersion period.

On the other hand, the Cd values of the specimens immersed in a 15% NaCl solution were significantly higher than those of the samples immersed in distilled water and 0.9% NaCl solution, which was mainly caused by the absorption and diffusion of Na^+ and Cl^- ions into the cement. Although the increment in the Cd values increased monotonically with increasing immersion time, the trend could be separated into three stages based on the following potential mechanism. In the initial stage, the Cd increased with increasing immersion time from 0.5 to 3 h. In this stage, the increase was caused by the absorption and diffusion of Na^+ and Cl^- with water. The next stage was the plateau stage to 1 day. In this phase, water was saturated with the cement, which suppressed the material transfer from the perimeter to the center. With the change in the material transfer direction, the release of ions in the cement was promoted and canceled the effect of the absorption and diffusion of Na^+ and Cl^- . Finally, at the third stage, an abrupt increase in the Cd values with increasing immersion time was observed. After the release of ions initially contained in the cement converged to zero, the absorption and diffusion of Na^+ and Cl^- were accelerated. To clarify the above hypothesis, further research on material transfer in cement and its effects on electrical conductivity are required.

Interestingly, although the Cd values of the specimen immersed in 15% NaCl solution for 1 day were similar to those immersed for 0.5 and 7 days, the σ_s reduction of specimen immersed for 1 day was smaller. This difference suggested that CA was less effective to the specimen immersed for 1 day than those immersed for 0.5 and 7 days. This non-linear correlation between the Cd value and σ_s reduction was possibly caused by the following mechanism: Because the Na^+ and Cl^- ions from the liquid diffused from the interface into the cement, the concentrations of Na^+ and Cl^- in the cement near the perimeter were higher than those near the center of the bonding area. The higher concentrations of Na^+ and Cl^- ion were effective in increasing the electrical conductivity and charge density, which enhanced the increment of

the Cd value in specimens immersed in a 15% NaCl solution. Higher Cd values are expected to yield lower σ_s . However, such values were observed only at the areas near the perimeter; thus, the areas contributing to the reduction of σ_s were limited. Simultaneously, with the release of ions, the Cd value of cement near the center of the bonding area was reduced, and, thus, the σ_s reduction near the center decreased. Because of these two changes in σ_s reduction, the minimum σ_s reduction was observed in the specimen immersed for 1 day. With increasing immersion time, Na^+ and Cl^- diffused to the center of the bonding area; thus, the area of higher Cd value and lower σ_s expanded all the way to the bonding area, and the reduction in σ_s of the specimen increased.

As can be seen in Fig. 1, the effect of immersion in 15% NaCl solution on the electrical conductivity of the specimens was larger than that of the immersion in distilled water and 0.9% NaCl solution. However, the effect of the Cd on σ_s reduction in the specimens immersed in a 15% NaCl solution was smaller than that in the samples immersed in distilled water and 0.9% NaCl solution, as shown in Fig. 3. To clarify the effect of NaCl concentration quantitatively, we performed a multiple regression analysis of the data shown in Fig. 3. The correlation between σ_s and the Cd and C_{NaCl} is given as:

$$\sigma_s = (-6.48 \times Cd) + (0.250 \times C_{\text{NaCl}}) + 15.1, \text{ adjusted } R^2 = 0.451$$

where C_{NaCl} is the NaCl concentration of the immersion liquid.

This correlation indicates that σ_s showed a slight but positive dependence on C_{NaCl} , that is, with increasing C_{NaCl} , the σ_s possibly showed slight increase. Therefore, CA was less effective for the debonding on-demand property of the cement immersed in NaCl solutions with higher concentrations.

Composition and concentration of immersion solution

In this section, the effect of the NaCl electrolyte and its concentration on the change in the electrical conductivity and σ_s of the specimen is discussed. The 0.9% NaCl solution used in this study was a model of saliva, and indicated the effectiveness of retaining the electrical debonding on-demand property. However, the actual electrolytes and concentrations of solutions in the oral cavity are more complicated. Even when limited to the most common solution existing in the oral cavity, the saliva, electrolytes, and their concentrations are not constant, and many types of artificial saliva have been utilized¹⁵⁾. The speed of the absorption of an ion to RMGIC depends both on the nature of the ion and its concentration in the solution, and the diffusion speed of an ion in the RMGIC depends on the element. In addition, the electrochemical reaction at the interface between RMGIC and Ti varies with changes in the electrolyte and the ion concentration in the RMGIC. Therefore, to simulate the effect of liquids in the oral cavity on the electrical shear bond strength reduction property, more detailed research using various types of

solutions and artificial saliva should be done.

On the other hand, the 15% NaCl solution was used to simulate liquid seasonings, such as soy sauce, which is safe in the oral cavity for a short time. For actual clinical applications, the cement in the oral cavity should be covered by a gel containing NaCl, similar to the tooth bleaching procedure using a tray¹⁶⁾. However, the required immersion time to enhance the σ_s reduction was excessively long for clinical application. After obtaining the minimum σ_s reduction value after 1 day of immersion, an additional 27 days was required to achieve a significant increase in σ_s reduction. Briefly, immersion in a higher NaCl solution was not effective for maintaining the electrical debonding on-demand property.

CONCLUSIONS

RMGICs were immersed in distilled water, and 0.9 and 15% NaCl solutions over different durations, and their properties were evaluated. The following conclusions were drawn.

1. RMGICs immersed in NaCl solutions showed enhanced the electrical conductivities relative to those immersed in distilled water with increasing immersion period.
2. The RMGIC sample immersed in 0.9% NaCl solution exhibited larger σ_s reduction than the sample immersed in distilled water after the application of current.
3. The σ_s of RMGIC immersed in a 15% NaCl solution exhibited the smallest reduction after 1 day immersion. Beyond that duration, the reduction increased with increasing immersion period.
4. The debonding interface was changed from the cathode to the anode after 28 days immersion in 15% NaCl solution, indicating that the electrochemical reaction on the electrode could be changed by the absorption and/or release of ions in the RMGIC in the oral cavity; thus, indicating that the debonding on-demand property could also be tuned.

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