INTRODUCTION

New and advanced dental cements exhibit superior properties compared to those of conventional materials\(^1\); especially, the improvement in the bonding strength gives patients the assurance of a better durability of their dental devices. However, the high bonding strength occasionally causes difficulties in the removal of the devices, which is needed in several circumstances: the removal of restorations is necessary for treating the patient from the incidence of secondary caries; removal of orthodontic brackets is required after the completion of an orthodontic treatment; and the removal of dental implant abutments is appropriate for the periodical maintenance of cement-retained dental implants.

Furthermore, excessive force or vibration is required for the removal of these devices, which occasionally leads to damages to the enamel, dentin, and teeth roots. Hence, the use of a “smart” dental cement exhibiting an adjustable bonding strength which is controllable on-demand is required in order to simultaneously have strong bonding and easy debonding.

Adhesives for engineering applications exhibiting easy on-demand debonding have been developed and released\(^2\). In contrast, the development of such adhesives for dental applications has only just started and has only been reported recently\(^3-7\). Many of these adhesives use heat as a trigger to reduce the bonding strength, taking advantage of softening, melting, or bubbling of the adhesives at high temperatures\(^8\). However, heating of adhesives in the oral cavity has the potential risk of damaging to the oral mucosa. Thus, an electric current has been adopted as the trigger in this work. We have developed a prototype resin-modified glass-ionomer-cement (RMGIC) with an ionic liquid (IL) and validated the concept of a “dental cement debonding on-demand in response to an electric current trigger” in a previous paper\(^9\).

KEYWORDS: Glass-ionomer-cement, Shear bonding strength, Ionic-liquid, Electric conductivity, Smart adhesive

MATERIALS AND METHODS

Prototype cement preparation

The IL used in this study was tris(2-hydroxyethyl) methyammonium methylsulfate (Sigma-Aldrich
Table 1 Composition of the RMGIC utilized in this study (data obtained from manufacturer safety data sheets)

| Paste A                  | Ingredient                        | mass% |
|-------------------------|-----------------------------------|-------|
|                         | Silane treated glass              | 70–80 |
|                         | Water                             | 10–20 |
|                         | 2-hydroxyethyl methacrylate (HEMA)| <10   |
|                         | Silane treated silica             | <2    |
|                         | 4-(dimethyamino)-benzeneethanol   | <1    |
|                         | Titanium dioxide                  | <0.5  |

| Paste B                  | Ingredient                        | mass% |
|-------------------------|-----------------------------------|-------|
|                         | Silane treated ceramic            | 30–40 |
|                         | 2-hydroxyethyl methacrylate (HEMA)| 10–30 |
|                         | Copolymer of acrylic and itaconic| 10–30 |
|                         | acids                              |       |
|                         | Water                             | 5–15  |
|                         | Glycerol 1,3 dimethacrylate       | 1–5   |
|                         | Potassium diposphate              | 1–5   |
|                         | Potassium persulfate              | 1–5   |
|                         | 2,6-di-tert-butyl-p-cresol (BHT)  | <0.5  |
|                         | Ethylene dimethacrylate (EGDMA)   | <0.5  |

Japan, Tokyo, Japan), the same one employed in the previous study\(^9\). It was considered suitable for the preparation of prototype cements due to its low toxicity and the absence of skin irritation. A commercial RMGIC (RelyX\textsuperscript{TM} Luting Plus, 3M Japan Limited, Tokyo, Japan) was utilized as the base cement. The composition of the RMGIC is presented in Table 1. The IL mixing ratios (wt%) were 0% (RX0) and 10% (RX10). The IL and two cement pastes (a base paste and a catalyst paste) were mixed simultaneously using a plastic sterile spatula on a mixing paper.

**Specimen preparation for the evaluation of the shear bond strength**

Dental cements are mainly utilized for the bonding of restorative materials to the dentin or the enamel. Thus, the bonding strength between these restorative materials and the dentin/enamel should be evaluated for perspective clinical applications. However, in this study, only the bonding strength between two metals was evaluated, because the objective was to clarify the effects of water immersion on the electrical properties and bonding properties of the prototype cement and to validate bonding strength reduction upon current application (CA). In our previous study\(^9\), Cu was adopted as the adherend material because of its high electric conductivity. In contrast, Ti was adopted in this study because the corrosion resistance of Cu in water is inadequate.

Two Ti rods (ø8×10 mm and ø20×10 mm (diameter×length)) were used to assemble the specimens. The cross section of the rods was sanded using 600 grit SiC waterproof paper, and then the circular bonding area of both rods was roughened by sandblasting (alumina particles of 50 µm diameter, sandblasting pressure of 0.4 MPa). After uniformity of the surface roughness 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 ø8 mm Ti rod, and it was set at the center of the ø20 mm Ti rod. The pair of rods were pressed at a pressure of 20 g/mm\(^2\) for 5 min and the excess cement was removed manually. Thirty min after bonding, the specimens subject to water immersion were left into distilled water and then kept either for 1 day, 7 days, or 14 days, and the specimens not subject to water immersion were stored in air for 24 h (immersion for 0 day). Distilled water (100 mL for one sample, 37°C) was exchanged every 3.5 days.

**CA to the specimens**

Direct currents of 19 V were applied for 30 s on the specimens in air using a programmable power supply (Type 7651, Yokogawa Electric, Tokyo, Japan). These conditions were set to be equal to those for the removal of ElectRelease\textsuperscript{TM} \(^11\). The ø20 mm rod was set as the anode. The changes in current with time were recorded using a digital multimeter (Type TY720, Yokogawa Electric), and the total charge density applied on the specimens was calculated from the time-integrated current values.
**Bonding strength evaluation**

A universal testing machine (AGS-500A, Shimadzu, Kyoto, Japan) was utilized for the evaluation of the shear bond strength, $\sigma_s$. The specimen setting in the compression test jig is schematically shown in Fig. 1. The cross head speed was set to 0.5 mm/min. $\sigma_s$ was calculated as follows:

$$\sigma_s = \frac{L}{S} \quad (1)$$

where $\sigma_s$ is the shear bond strength, $L$ is the load at fracture, and $S$ is the bonding area (50.24 mm$^2$ in this study). To clarify the effects of CA on $\sigma_s$ values, the specimens were evaluated with and without CA.

The total specimen preparation and $\sigma_s$ evaluation flow is summarized in Fig. 2, and the number of the specimens of each group are indicated in Fig. 2.

**Fracture surface observation**

After $\sigma_s$ evaluation, the fracture surfaces of the specimens were photographed using digital cameras with automatic white-balance and exposure settings.

**Statistical evaluation**

The data for $\sigma_s$ was statistically analyzed using the Steel-Dwass test performed by the EZR software (Saitama Medical Center, Jichi Medical University, Japan) to compare the mean values from different groups. Statistical significance was accepted at a confidence level of 0.05 or 0.01.

The $\sigma_s$ measurements data was also investigated using a two-parameter Weibull distribution analysis (Eq. 2) applying the Bemard’s median rank method (Eq. 3).

$$P = 1 - \exp[-\left(\frac{\sigma_s}{\sigma_0}\right)^m]\quad (2)$$

$$P = \frac{i - 0.3}{n + 0.4} \quad (3)$$

Where $P$ is the cumulative probability of fracture, $\sigma_s$ is the shear bond strength, $\sigma_0$ is the scale parameter, $m$ is the Weibull modulus (shape parameter), $i$ indicates that a $\sigma_s$ value ranks the $i$th in the ascending order of magnitude for $\sigma_s$ and $n$ is the number of total data point. Linear regression analysis was carried out to Weibull plots of ln[ln(1/1–$P$)] vs. ln($\sigma_s$), then, the Weibull modulus and scale parameter have been estimated.
RESULTS

Charge density

Figure 3 shows the effect of the duration of immersion in distilled water on charge density with CA, and Table 2 shows the statistical analysis of these results. The charge density average for the RX0 specimens subject to water immersion was significantly higher than that of the specimens not subjected to water immersion, however, there was no significant effect of the immersion period on charge density. Although the immersion time did not have a significant effect on the charge density, the trend of the average values for multiple samples potentially shows reduction and saturation with increasing immersion time from 1 day to 14 days. In contrast, the charge densities of the RX10 specimens with and without water immersion did not show a significant difference.

Although there was no significant influence of the immersion period, the average value potentially shows a higher value after the first day, which decreased and eventually reached a plateau with increased immersion time from 1 day to 14 days. A comparison of the results from the RX0 specimens and the RX10 specimens indicates that the RX10 specimens not exposed to water and those subject to 7 days and 14 days of immersions showed a significantly higher charge density average compared to those of the RX0 specimens.

Shear bond strength

Figure 4 shows the effect of immersion time on $\sigma_s$, and Table 3 shows the statistical analysis of these findings. The immersion time did not have a significant effect on $\sigma_s$ for both the RX0 specimens and the RX10 specimens without CA. In contrast, $\sigma_s$ of the RX0 specimens and the RX10 specimens after 1 day immersion with CA showed a significant decrease compared to the case where the specimens were not immersed. In particular, for the RX10 specimens after 1 day immersion and CA, the $\sigma_s$ reached the lowest value in this study. The $\sigma_s$ of the RX0 specimens and the RX10 specimens with CA increased significantly with immersion from day 1 to day 7. The average value of the RX10 specimens with CA

Table 2  Statistical analysis of the charge density with CA

| specimen | immersion period in distilled water (day) | Significance |
|----------|------------------------------------------|--------------|
| RX0      | 0                                        | $p<0.05$     |
|          | 1                                        | $p<0.01$     |
|          | 7                                        |              |
|          | 14                                       |              |
| RX10     | 0                                        | $p<0.05$     |
|          | 1                                        | $p<0.01$     |
|          | 7                                        |              |
|          | 14                                       |              |
showed an initial increase and subsequent saturation with increasing immersion time from 1 day to 14 days, and that of the RX0 specimens showed a similar trend. Comparison of $\sigma_s$ values with and without CA indicates that for RX10 specimens, the average $\sigma_s$ with CA was significantly lower than that without CA; however, for the RX0 specimens with CA subject to 7 days and 14 days immersion, $\sigma_s$ did not show a significant decrease compared to those without CA. Besides, without CA, $\sigma_s$ of the RX10 specimens was significantly lower than that of the RX0 specimens, and also with CA, $\sigma_s$ of the RX10 specimens was significantly lower than that of the RX0 specimens ($p<0.01$, not indicated in Table 3).

Figures 5 and 6 show the Weibull plots of $\sigma_s$ of the RX0 specimens with and without CA, and that of the RX10 specimens with and without CA, respectively. The Weibull modulus and scale parameter estimated are shown in Table 3. The Weibull modulus of the RX0 specimens without CA had the highest value in this study. Both the RX0 and RX10 specimens with 1 day immersion with CA exhibited the smallest Weibull modulus. Note that the RX10 specimens subject to 1 day immersion with CA showed an extremely small Weibull modulus and the Weibull plot showed a non-linear trend.

Effect of charge density on the shear bond strength
Figure 7 shows the effect of charge density on the $\sigma_s$ for the RX0 specimens and the RX10 specimens. As seen, $\sigma_s$ decreased linearly with increasing charge density from 0 to approximately 1 mC/mm².

Fracture surface after the shear bond strength test
Figure 8 shows the typical fracture surfaces of Ti rods of the RX0 specimens and the RX10 specimens after 1 day of immersion with and without CA, after they were subjected to the shear bond strength test. Both the Ti surface and cement residues were observed without CA on the surface of many specimens, which indicates that the fracture was mixed mode; however, few specimens showed interface fracture. In contrast, no cement residue was observed on the anode of many specimens with CA, indicating that the fracture was interfacial at the anode. However, few RX0 specimens showed mixed mode fracture. Correlation between the shear bonding strength and fracture mode was not clear in this research.

DISCUSSION
Changes in charge density with increasing immersion period
The base RMGIC for the prototype cement before curing contains water-soluble polymers and polymerizable monomers, glass fillers, tartaric acid, and water. Thus, the base RMGIC can exhibit electric conductivity in case
The legends indicate immersion period in distilled water.

Fig. 6 Weibull plot of the shear bonding strength of RX10 specimen with and without CA. The legends indicate immersion period in distilled water.

Fig. 7 Correlation between the charge density and the shear bonding strength.

if tartaric acid is not consumed completely in the acid-base reaction occurring during the curing phase and if water is not completely consumed or released from the cement. Figure 3 indicates that the RX0 specimens without immersion showed only small charge density values, and the RX0 specimens after 1 day immersion showed a significant increase in charge density. This fact suggests that our RMGIC is not an electrical insulator but a weak electrical conductor, and that it can increase its electric conductivity with water absorption. The charge density average value for the RX0 specimens decreased with increased immersion period from day 1 to day 7. The difference was not significant; however, the average value showed a decreasing trend from 1 day to 7 days of immersion, and then saturated at a higher value than those of the same samples without immersion. This decrease was potentially caused by a release of tartaric acid from the specimen. Therefore, we would expect that the charge density keeps decreasing with increased immersion period. To verify that the charge
Fig. 8 Photographs and their schematic images of fracture surfaces on the RX0 specimen and RX10 specimen with 1 day immersion with and without CA after the shear bonding strength test. Black area in the schematic images indicates the cement residue.

Changes in shear bond strength after CA and with increasing immersion periods

Figure 4 shows that the decrease of $\sigma_s$ for the RX0 specimens due to CA was not necessarily significant, however the average value decreased slightly due to CA, regardless. The decrease of $\sigma_s$ for the RX0 specimens after 1 day immersion due to CA was the largest, which is comparable to the large charge density change in the RX0 specimens after 1 day of immersion. Note that the RMGICs without IL possibly show an on-demand debonding property with CA, based on their electric conductivity. This finding reminds us that an applied Galvanic current potentially has the risk of reducing the density decrease really plateaus, an evaluation of the effect of a longer immersion time for the RX0 specimens is required.

With the IL, the RX10 specimens showed a significantly higher charge density average than did the RX0 specimens, indicating that IL addition is effective to increase the electric conductivity of an RMGIC. Although the difference was not significant, the charge density value increased after 1 day of immersion and then decreased over 7 days and 14 days. The dependence of charge density for the RX10 specimens on immersion time was similar to that of the RX0 specimens. The increase was also caused by water absorption, while the decrease was also caused by a release of tartaric acid and/or IL from the specimen. The increment in the charge density value from 0 day immersion to 1 day immersion of the RX10 specimens was smaller than that of the RX0 specimens, but the reason for this was not clarified in this study. Note that the charge density values of the RX10 specimens with immersion periods longer than 7 days were nearly equal to those of the samples without immersion, demonstrating that the RX10 specimens hardly lose any electric conductivity solely because of the effect of distilled water.

Changes in shear bond strength after CA and with increasing immersion periods

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bonding strength between metal restorations and dentin in the oral cavity, resulting in a premature and uncontrolled debonding of the restorations. However, the decrease of $\sigma_s$ for the RX0 specimens shows a decrease with increasing immersion period, promising an alleviation of the risk over time after bonding. Figure 7 shows that the average $\sigma_s$ for the RX0 monotonously decreased with increasing charge density. This dependence was similar to that in our previous study\textsuperscript{20}, however, average $\sigma_s$ for the RX0 samples showed a linear decrease in these experiments, while it showed an exponential decrease in the previous study. The reason of this mismatch was not yet clarified. One possible reason is that the bonding metal in this study was titanium, while copper was used in the previous experiments. A linear and monotonous decrease of $\sigma_s$ was also observed for the RX10 specimens, and the dependence of the $\sigma_s$ on charge density was also similar to the other specimens.

Figure 4 shows that $\sigma_s$ for the RX10 specimens not subject to immersion without CA was significantly lower than that of the RX0 specimens without CA, indicating that 10% IL in the material was excessive and reduced $\sigma_s$. We have evaluated specimens with low IL and found $\sigma_s$ to increase with decreasing IL content, and on the other hand, electric conductivity decreased with decreasing IL content. Since the decrease in electric conductivity requires longer periods to perform on-demand debonding\textsuperscript{20}, optimization of the IL content to achieve both optimal initial $\sigma_s$ and appropriate periods to perform on-demand debonding is required.

Such as the decrease of $\sigma_s$ in the RX0 specimens, the decrease of $\sigma_s$ value for the RX10 specimens after 1 day immersion due to CA was very substantial, which is compatible with the large difference in charge density in the RX10 specimens in the same conditions. The change in $\sigma_s$ for the RX10 specimens with CA was found to be larger than that of the RX0 specimens subject to the same immersion periods in this study, which is also compatible with the results that a larger change in charge density value is observed for the RX10 specimens compared to the RX0 specimens. This result indicates that IL addition to RMGICs is effective to reduce the $\sigma_s$ with CA. The decrease of $\sigma_s$ slowed down from 1 day immersion to 7 days immersion and 14 days immersion; however, the decrease was completely saturated after 7 days of immersion. This fact suggests that the imperfect permanence of ion content in distilled water decreases the electric conductivity of the prototype cement and sequentially inhibits the reduction of $\sigma_s$ with CA, but this inhibition effect will not be rapid.

Weibull plots of $\sigma_s$ values shown in Figs. 5 and 6 did not reveal remarkable differences between different cases, except for $\sigma_s$ values of the RX0 specimens with 1 day of immersion with CA. The RX0 specimens not subject to immersion without CA demonstrated the largest Weibull modulus, namely, the $\sigma_s$ value distribution for the RX0 specimens without immersion and without CA was the smallest. The property of the other specimens changed due to a manual mixing of the IL into the RMGIC and subsequent immersion in distilled water, as well as CA. These alterations potentially enlarged the distribution of $\sigma_s$ values; in particular, manually obtaining a homogeneous mixture of IL and RMGIC was difficult because of the high viscosity of the IL chosen. Note that the Weibull plot for the $\sigma_s$ values of the RX0 specimens with CA after 1 day of immersion was apparently different from those of the other plots; it did not show a linear trend and potentially showed a minimum $\sigma_s$ value at approximately 0.1 MPa. These results suggest that a truncated Weibull distribution analysis (Eq. 4) is more appropriate in this situation\textsuperscript{16}.

\[ P = 1 - \exp\left(-\left(\frac{\sigma_s - \sigma_{0}}{\sigma_{0}}\right)^{m}\right) \]  

Where $P$, $\sigma_s$, and $\sigma_0$ are the same as those in Eq. 2, and $t$ is the truncation limit. The truncation limit of $\sigma_s$ indicates the minimum $\sigma_s$ value at which the specimen was found to be durable to the shear bonding strength test in our experiments. Under the truncation limit, the bonding in the specimen was damaged, and debonding happened during the experimental procedure of CA, or while setting the sample in the test apparatus. Thus, the data acquisition of very low $\sigma_s$ values was impossible, and these data was ignored in the two-parameter Weibull distribution analysis. Figure 9 shows the truncated Weibull distribution analysis with a scale parameter of 0.01 MPa, a Weibull modulus (shape parameter) of 0.33, and a truncated limit of 0.08 MPa. We can see that it shows a more accurate representation of the behavior of the RX10 specimens with 1 day immersion with CA. This consideration suggests that two-parameter Weibull analysis of the entire data set, including lower $\sigma_s$ value data not acquired in this research, will indicate smaller scale parameters and smaller Weibull parameters. Conversely, the two-parameter Weibull analysis result
overestimate the value of $\sigma$, and underestimate the distribution of $\sigma$ values for the RX10 specimens with 1 day immersion with CA.

**Effects of CA on the bonding between the cement and titanium**

Figure 8 shows that an interface fracture with CA was observed only on the anode. This result coincides with the fracture pattern obtained in the previous study. Therefore, the reason for the decrease of $\sigma$ was possibly similar: Ti ions release, and/or a potential Ti compounds formation damages the interfacial bonding between the cement and the anode. Thus, a large charge density should promote reduction in $\sigma$.

**Future work**

It is widely recognized that many RMGIC-based products today show not only the release of fluoride ions, but also their recharge. This phenomenon suggests that RMGICs potentially absorb the ions contained in the oral cavity, e.g. saliva, drinks and foods containing salts, resulting in a maintenance of a somewhat high electric conductivity for long periods. Thus, in our next investigation, the prototype cement explored in this research will be immersed in saline or artificial saliva, and the changes in charge density and decrease in bonding strength with CA will be evaluated.

**CONCLUSIONS**

RMGICs with or without ILs were produced and immersed in distilled water over different time scales, and their properties were evaluated. The following conclusions can be drawn:

1. RMGICs without ILs exhibited significant electric conductivity after immersion, and a significant bonding strength decrease after CA.

2. RMGICs with ILs exhibited larger electric conductivity than RMGICs without ILs after immersion, and more significant bonding strength decrease after CA.

3. The electric conductivity of RMGICs both with and without ILs increased after 1 day of immersion, and then decreased after 7 days and 14 days immersions. However, the values for samples tested after 14 days immersion were similar to, or larger, than those before immersion.

4. The bonding strengths of the RMGICs with ILs after CA exhibited significant decreases after 14 days immersion, and eventually reached a plateau.

These results suggest that RMGICs with ILs are potential “smart” dental cements and are not subject to substantial impairment of their properties after immersion in the liquids of the oral cavity.

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