INTRODUCTION

Nowadays, zirconia-based restorations have been used widely in dentistry thanks to their superior mechanical properties, biocompatibility, chemical stability, and optical properties1-4. Full contour restorations made by highly translucent zirconia ceramics are becoming more prevalent due to esthetically pleasing5). However, because of glass-free polycrystalline microstructure and chemical inertness, etching with hydrofluoric acid or silanization has not been effective on zirconia surfaces3,6) and thus many studies have focused on achieving strong and durable bonding to zirconia ceramics. To improve bonding efficacy of resin cement to zirconia ceramics, several mechanical pre-treatments such as air abrasion with aluminum oxide particles7), tribochemical silica coating6,8,9), laser irradiation10), chemical etching11), ceramic coating12), and also chemical pre-treatment with primers containing functional monomers1,13-15) have been reported.

According to systematic review and meta-analysis studies, both mechanical and chemical pre-treatments are essential to obtain durable bonding to zirconia15-17). Kern and Wegner6) reported that sandblasting with Al2O3 and resin cements containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) as a monomer is essential to achieve strong and durable bonding to zirconia ceramics. MDP currently is the most widely used functional monomers and promote chemical bond to zirconia and potentially create a durable bond14,15,17). Despite the progress made with MDP primer, the bonding to zirconia is still prone to hydrolytic degradation18,19).

4-methacryloxyethyl trimellitate anhydride (4-META) adhesive resins as a carboxylic monomers have been reported in the dental literature for over three decades. 4-META which was invented in 1978 has been shown to be effective in improving bonding of non-precious metals and metal oxides20). Super Bond C&B cement (Sun Medical, Shiga, Japan) that contained 4-META, methyl methacrylate (MMA) and tri-n-butyl borane (TBB), has shown high bond strength with non-precious metals20) and zirconia21). 4-methacryloyloxyethyl trimellitic acid (4-MET) is an active form of 4-META that after adding water to 4-META powder, quick hydrolysis reaction will occur to form 4-MET22). However, there is lack of information as to differences in bonding efficacy between 4-META and 4-MET to highly translucent zirconia ceramics.

The purpose of this study was therefore to investigate the effect of carboxyl-based monomers including 4-META and 4-MET on the bond durability of TBB-initiated resin to highly translucent zirconia ceramics. The null hypothesis tested was that surface pre-treatment with carboxylate monomers does not achieve strong and durable bonding to zirconia ceramics.

The effect of carboxyl-based monomers on resin bonding to highly translucent zirconia ceramics

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The purpose of this study was to evaluate the bonding performance of carboxyl-based monomers in the priming agents to highly translucent zirconia. Highly translucent zirconia disk-shaped specimens of yttria-partially stabilized zirconia (Y-PSZ) (Katana HT zirconia) after sandblasting, were assigned to 5 groups according to the chemical pretreatment: no-treatment (Con), PZ Primer (PZP), PZ primer liquid A+MMA (PZA, as MDP primer), Super-Bond liquid (SBL, as 4-META primer) and experimental 5% 4-MET primer (MET). After priming, stainless steel rods were bonded to the zirconia specimens with TBB-initiated resin. The tensile bond strength test was performed after storage for 24 h, 30, and 150 days in 37°C water. The Weibull moduli decreased in all groups except PZA after 150 days. Superior bond strength observed in PZA and MET groups after aging. Application of carboxyl-based monomers were found to be effective in adhesion to highly translucent zirconia ceramics.

Keywords: Highly translucent zirconia, Bond strength, Carboxyl-based monomer

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improve the bond strength of TBB-initiated resin to highly translucent zirconia.

MATERIALS AND METHODS

The materials tested in this study are listed in Table 1. Fully-sintered zirconia disk-shaped specimens (12 mm diameter; 5 mm thickness) of highly translucent zirconia were fabricated with yttria-partially stabilized zirconia (Y-PSZ; Katana HT zirconia, Kuraray Noritake Dental, Tokyo, Japan) were used as the bonding substrate. Four primers with different functional monomers which contain at least one adhesive functional monomer such as MDP, trimethoxysilylpropyl methacrylate (3-TMSPMA), 4-META, and 4-MET were used in this study.

An MMA-based self-polymerizing resin initiated with partially oxidized TBB was used as a luting agent. The TBB initiator and the powder component of this material were the same as those of Super-Bond C&B (Sun Medical). A 99.8% MMA (Sun Medical), which did not contain any functional monomers, was chosen as the monomer liquid of the MMA-TBB resin used in this study because the purpose of the current study was to evaluate the functional monomers in the priming agents.

The complete study design is schematically explained in Fig. 1. The surfaces of the specimens were wet-polished using 600-grid SiC paper and then the bonding surfaces were blasted with 50 µm alumina particles (Aluminous powder 50 µm, Kulzer Japan, Tokyo, Japan) from a distance of 10 mm for 20 s at 0.2 MPa, using a sandblasting device (Renfert Basic eco, Renfert, Harlingen, Germany). Next, specimens were ultrasonically cleaned with ultrapure water for 5 min and 99% ethanol for 2 min. They were randomly assigned to one of the following 5 groups ($n=14–15$). (1) Group Con: no additional chemical pre-treatment; (2) Group PZP: mixed PZ Primer liquid A and B (1:1) (Sun Medical) and applied; (3) Group PZA: mixed PZ primer liquid A and MMA (Sun Medical) (1:1) and applied; (4) Group SBL: applied Super-Bond monomer (Sun Medical); (5) Group

| Material                  | Manufacturer                      | Type of material | Composition               | Application procedure                                                                 |
|---------------------------|-----------------------------------|------------------|---------------------------|--------------------------------------------------------------------------------------|
| KATANA HT zirconia        | Kuraray Noritake Dental, Tokyo, Japan | Y-PSZ            | 94.4% ZrO$_2$, 5.4% Y$_2$O$_3$ | 1. Blast the surface from a distance of 10 mm for 20 s at 0.2 MPa pressure. 2. Ultra-sonically clean in ultrapure water for 5 min and ethanol 2 min. |
| Alumina (Al$_2$O$_3$)     | Kulzer Japan, Tokyo, Japan         | Alumina powder   | 50 µm Al$_2$O$_3$         |                                                                                      |
| Alloy Primer              | Kuraray Noritake Dental            | Metal primer     | MDP, VBATDT, acetone      | Coat primer on the surface of the alumina-blasted metal rods.                        |
| Super-Bond PZ Primer      | Sun Medical, Shiga, Japan          | primer           | Liquid A: MDP, MMA        | Apply primer for 10 s, then thoroughly air drying.                                    |
| Super-Bond monomer        | Sun Medical                        | primer           | 4-META, MMA               | Apply primer for 10 s, then thoroughly air drying.                                    |
| 4-MET experimental primer | Sun Medical                        | primer           | 5% 4-MET, MMA             | Apply primer for 10 s, then thoroughly air drying.                                    |
| Super-Bond powder         | Sun Medical                        | MMA-based resin  | PMMA                      | 1. Brush loading on the zirconia disk. 2. and remove the excess cement and fix with a ratchet bar clamp for 30 min |
| Super-Bond Catalyst V     | Sun Medical                        | Initiator        | TBB                       |                                                                                      |
| MMA                       | Sun Medical                        | Monomer          | MMA 99.8%                 |                                                                                      |

MDP: 10-methacryloxydecyl dihydrogen phosphate; VBATDT: 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-trizaine-2,4-dithiol; MMA: methyl methacrylate; 3-TMSPMA: 3-trimethoxysilylpropyl methacrylate; 4-META: 4-methacryloyloxyethyl trimellitate anhydride; 4-MET: 4-methacryloyloxyethyl trimellitic acid; PMMA: polymethyl methacrylate; TBB: tri-n-butyl borane
Table 2  Experimental groups of chemical surface treatment

| Groups | Primer                                | Functional monomer |
|--------|---------------------------------------|--------------------|
| Con    | No-treatment                          | —                  |
| PZP    | Liquid A + Liquid B of PZ primer       | MDP+3-TMSPMA       |
| PZA    | Liquid A of PZ primer + MMA            | MDP                |
| SBL    | Super Bond Monomer                    | 4-META             |
| MET    | Experimental 4-MET primer             | 4-MET              |

MET: applied experimental 5% 4-MET primer (Sun Medical). Table 2 indicates the experimental groups of chemical surface treatment.

A piece of aluminum tape with a circular hole of 4 mm diameter and 100 µm thickness was placed on the specimen surface to demarcate the bonding area. Stainless rods (8 mm diameter, 30 mm height) were used as adherend. Stainless rods were sandblasted for 30 min using 125 µm alumina particles (Vario Jet, Renfert) and then were ultrasonically cleaned with ultrapure water for 10 min. Next, a metal primer (Alloy Primer, Kuraray Noritake Dental) was applied on the surface of the stainless rods. After priming, alloy primer-treated stainless steel rods were bonded to the zirconia specimens with MMA-based resin cement initiated with TBB (Super Bond C&B powder, catalyst and 99.8% MMA liquid) by using a brush-dip technique under custom-made jig.

The specimens were stored at room temperature for 30 min, followed by water at 37°C for 24 h, 30 days and 150 days. Tensile bond strength (TBS) was measured using a universal testing machine (AGS-J, Shimadzu, Kyoto, Japan) at a crosshead speed of 2 mm/min. The TBS results were statistically analyzed using Weibull analysis with R3.1 and Abrem package (R Foundation for statistical computing, Vienna, Austria). Pivotal confidence bounds were calculated using the Monte Carlo simulation. All tests were performed at a significance level of α=0.05.

After the tensile bond testing, failure modes were examined under a stereomicroscope (SMZ1000, Nikon, Tokyo, Japan) at a magnification of 35×. The failure modes were classified into the following three categories: (1) adhesive failure at the interface between zirconia surface and resin cement, (2) cohesive failure only within the resin cement, and (3) mixed failure combination of cohesive and adhesive failure.

RESULTS

The results from the Weibull analysis are graphically presented in Fig. 2. Table 3 summarizes the Weibull analysis for all specimen groups. After 150 days water storage, Weibull moduli of all the tested groups
Fig. 2 (a) Graph showing the Weibull analysis for all conditions under water for 24 h. The dotted lines represent 95% confidence interval. Significant difference was observed for PZA (MDP) group. (b) Graph showing the Weibull analysis for all conditions after storing in water for 30 days. The dotted lines represent 95% confidence interval. Control group (no-treatment) resulted in significantly lower bond strengths. (C) Graph showing the Weibull analysis for all conditions under water for 150 days. The dotted lines represent 95% confidence interval. Control group resulted in significantly lower bond strength than PZA (MDP) and MET (4-MET) groups.
Fig. 3 Results of the failure mode analysis. All specimens showed cohesive failure in PZP and PZA groups, whereas mixed failure was observed in MET and SBL groups. Control group showed adhesive failure in some specimens.

Table 3 The results of Weibull analysis

| Chemical pre-treatment | Aging¹ | N   | Ptf² | Shape (modulus)³ | Scale (B63.2)⁴ | 95% confidence level at B 63.2⁵ |
|------------------------|--------|-----|------|------------------|-----------------|-------------------------------|
| Con                    | 24h    | 15  | 0    | 4.01             | 21.48           | 16.5–22.6 (b)                 |
|                        | 30d    | 15  | 0    | 1.49             | 7.97            | 3.8–9.1 (c)                   |
|                        | 150d   | 15  | 0    | 1.07             | 6.86            | 2.5–8.3 (c)                   |
| PZP                    | 24h    | 14  | 0    | 5.72             | 21.16           | 17.4–22.0 (b)                 |
|                        | 30d    | 15  | 0    | 4.68             | 19.39           | 15.5–20.2 (b)                 |
|                        | 150d   | 14  | 0    | 3.87             | 10.04           | 7.5–10.6 (b,c)                |
| PZA                    | 24h    | 15  | 0    | 3.89             | 31.01           | 23.7–32.8 (a)                 |
|                        | 30d    | 15  | 0    | 6.83             | 21.15           | 18.1–21.8 (b)                 |
|                        | 150d   | 15  | 0    | 3.87             | 14.40           | 11.0–15.2 (b)                 |
| SBL                    | 24h    | 14  | 0    | 5.66             | 19.94           | 16.4–20.7 (b)                 |
|                        | 30d    | 15  | 0    | 7.56             | 16.80           | 14.6–17.2 (b)                 |
|                        | 150d   | 15  | 0    | 3.59             | 10.87           | 8.0–11.4 (b,c)                |
| MET                    | 24h    | 15  | 0    | 6.41             | 21.18           | 17.9–21.8 (b)                 |
|                        | 30d    | 15  | 0    | 4.10             | 19.97           | 15.3–21.0 (b)                 |
|                        | 150d   | 15  | 0    | 3.10             | 14.66           | 10.4–15.7 (b)                 |

1. Aging protocol with ‘24h’ referring to 24 storage in 37°C Milli-Q water, and ‘30d’ referring to 30 days and ‘150d’ referring to 150 day storage in 37°C Milli-Q water; 2. Pre-testing failure; 3. The higher the Weibull shape, the more reliable the treatment; 4. The higher the Weibull scale, the higher the bonding effectiveness; 5. Same letters within brackets indicate absence of statistical significance at B63.2.

The failure mode distribution is summarized in Fig. 3. All specimens showed cohesive failure in PZP and PZA groups, whereas mixed failure was observed in MET and SBL groups. Adhesive failure was observed in the control group.

DISCUSSION
In the present study, the bonding effectiveness of carboxyl-based monomers on resin bonding to highly translucent zirconia ceramics of a TBB-initiated resin
was evaluated. MDP based primers improved the bond strength of TBB-initiated resin to zirconia. Surface pre-treatment with carboxylic-based monomers such as 4-META and 4-MET showed no difference in bonding performance comparing with MDP based primers. Thus, the null hypothesis that surface pre-treatment with carboxylate monomers does not improve the bond strength of TBB-initiated resin to highly translucent zirconia was rejected.

In this study, a TBS test was used to evaluate the efficiency of bonding to zirconia ceramics. The most frequently applied method for the bond strength between resin cement and zirconia-based on a meta-analysis was shear bond strength test\textsuperscript{24}. This is likely due to its simple specimen preparation and the test. However, the shear bond strength test has repeatedly been documented to result in inhomogeneous stress distribution along the interface\textsuperscript{24}. On the other hand, despite the reliability of µTBS test compared to other test methods\textsuperscript{25}, specimen preparation for µTBS test especially on zirconia is difficult and may cause to damage zirconia ceramics. Therefore, TBS test was chosen in this study.

Thermal cycling and long-term water storage have been commonly used to simulate aging of resin bonds to ceramic. However, it has been shown that different bonding systems are influenced differently by these two parameters\textsuperscript{5,26}. In this study, long-term water storage was chosen for aging, because thermal cycling may increase bond strength MMA-resin cement\textsuperscript{27}. It could be attributed to the increased mobility of radicals, which would be expected to lead to additional polymerization during heating in the thermal cycling procedure\textsuperscript{28}.

The TBB-initiated resin used in this study did not contain any adhesive monomer. This composition enabled evaluation of the effects of functional monomers of the primers. In this study, four different primers were investigated. PZA group containing MDP alone showed the highest TBS at 24 h. This result is in agreement with previous studies which the application of a phosphate functional monomer (MDP) was effective in bond strength between resin-based luting agents and zirconia ceramics\textsuperscript{2,6}. Indeed, in the comparison between base metal alloys and metal oxides\textsuperscript{28}, including a study by Derand P and Derand T\textsuperscript{29} evaluated different surface treatments and resin cements and found that 4-META/MMA-TBB resin cement (Super bond C&B, Sun medical) exhibited the significantly highest bond strengths to zirconia ceramics regardless of surface treatment (silica coating, sandblasting, HF etching, or grinding with a diamond bur). Besides, Shimizu et al.\textsuperscript{30} evaluated the bonding efficacy of 4-META/MMA-TBB resin to highly translucent zirconia subjected to different surface treatments. They concluded that 4-META/MMA-TBB resin showed durable bond strength regardless of whether an MDP-containing primer was used. In the present study, two carboxyl-based monomers, 4-META in SBL group and 4-MET in MET group were employed. No significant difference was observed between primers containing MDP and those containing 4-META or 4-MET even after 150 days water storage. It could be due to carboxylic monomers may chemically adhere to zirconia. According to Shimoe et al.\textsuperscript{30}, in which the bonding between zirconia surfaces and functional monomers was investigated using X-ray photoelectron spectroscopy (XPS) and energy dispersive X-ray microanalysis, an increase in the [C] and [O] intensity peaks was obvious when the zirconia peaks on primed surfaces were masked off by absorbed primer films, denoting that 4-META chemically adheres to the zirconia surface. On the other hand, the difference between 4-META and MDP which 4-META has less adsorption than MDP was elucidated by an analysis of the atomic ratios of [C]. Also, Pilo et al.\textsuperscript{31} evaluated the changes in the surface chemistry of yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) induced by primer treatments with Z primer plus (Bisco, Schaumburg, IL, USA) or Z-Bond (Danville materials, San Ramon, CA, USA). They indicated primers formed carboxylate and phosphate salts on Y-TZP, promoting chemical interaction to zirconia. However, they also mentioned hydrolytic sensitivity of the carboxylate salts may affect the strength and durability of adhesive resin interfaces with Y-TZP. In the present study, failure mode analysis showed no adhesive or mixed failure in MDP containing groups (PZP and PZA), it might be due to better chemical interaction of MDP to zirconia surface.

However, Yagawa et al.\textsuperscript{32} concluded the application of priming agents containing MDP resulted in higher post-thermal cycling bond strengths than that of priming agents containing carboxylic monomer (4-META or MAC-10). This difference might be due to the difference in luting agents applied (Bis-GMA based composite cement versus TBB-initiated resin). Bond strength results might be not only affected by chemical adsorption of functional monomer but co-polymerization of those acidic functional monomers with resin cement itself.

On the other hand, it seems that due to containing the active form of 4-META, MET group might be
more effective than SBL. In literature, there is little information about the difference in the effect of 4-META and 4-MET on promoting bonding to highly translucent zirconia. The results need further studies to evaluate it.

Long-term water storage without mechanical aging may be a limitation of this study. In future studies, bonding durability of carboxylic monomers with different luting cements to highly translucent zirconia ceramics under mechanical aging should be considered.

CONCLUSION

The application of carboxyl-based monomers was found to be effective in achieving a durable bonding to highly translucent zirconia ceramics.

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