Combined effect of two-liquid silane-phosphate primer and single-liquid sodium sulfite primer on bonding between self-polymerizing resins and feldspathic ceramics

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The purpose of this study is to investigate the combined effect of two-liquid silane-phosphate primer and single-liquid sodium sulfite primer on bonding between self-polymerizing resins and feldspathic ceramics, and to promote the polymerization behavior of self-polymerizing resins at the bonding interface. The silane-phosphate primer (Super-Bond PZ Primer; PZ) and the sodium sulfite primer (Teeth Primer; TP) were used as the surface treatment agents for bonding the feldspathic ceramics and the self-polymerizing resins (MMA-TBB resin and 4-META/MMA-TBB resin). Combined PZ and TP showed high shear bond strengths. The peak of the differential scanning calorimetry curve was shown to occur early through the addition of TP. These results indicated that additional TP promoted the conversion in the initial polymerization of resin, and the firmly bond was obtained at the interface. PZ+TP treatment can be regarded as an effective treatment for a temporary splint used in teeth restoration.

Keywords: Feldspathic ceramics, Silane, Sodium sulfite, Temporary splint, Tri-n-butylborane

INTRODUCTION

For the purposes of teeth preservation, the protection of periodontal tissue, and the prevention of teeth movement, a temporary splint is an effective treatment when teeth mobility occurs from periodontal disease or dental trauma. Several methods exist for applying a temporary splint, although in many cases, an adhesive resin-based splint using a self-polymerizing resin is frequently used. When applying an adhesive resin splint, it is necessary to bond the moving teeth and the adjacent teeth surfaces with a resin. However, it is difficult to obtain a strong splint for teeth treated with ceramic restorations.

The bonding procedure between the feldspathic ceramics and the self-polymerizing resin has been established. When bonding feldspathic ceramics and a self-polymerizing resin, an alumina airborne-particle abrasion and etching with hydrofluoric acid are applied to obtain a micromechanical interlock, followed by silanization. A temporary splint is applied inside the mouth; therefore, it is impossible to apply an etching with hydrofluoric acid. In addition, alumina airborne-particle abrasion has a risk of volume loss of feldspathic ceramics. It is then necessary to achieve a chemical bond between feldspathic ceramics and a self-polymerizing resin without alumina airborne-particle abrasion or etching with hydrofluoric acid.

The self-polymerizing resin, 4-methacryloxyethyl trimellitate anhydride/methyl methacrylate-tri-n-butylborane (4-META/MMA-TBB), shows a high conversion at normal temperature, and the amount of residual monomer after polymerization is extremely low. Furthermore, because polymerization initiates from the bonding interface, that is the direction in which polymerization shrinkage is directed. For these reasons, it is possible to obtain a high bond strength between the feldspathic ceramics and the 4-META/MMA-TBB resin, which has been widely used as a luting agent and a temporary splint material.

The effect of a sodium sulfite primer has been reported to improve the bond strength between 4-META/MMA-TBB resin and enamel or dentin. These studies have speculated that a sodium sulfite primer affects the interfacial polymerization of the MMA-TBB resin. However, the combined effect of a silane-phosphate primer and a sodium sulfite primer on the bonding between the feldspathic ceramics and self-polymerizing resin has not yet been reported. Regarding a temporary splint, there are cases in which natural teeth have been splinted with a self-polymerizing resin, although there remains limited information regarding a temporary splint applied to a fixed dental prosthesis using ceramic materials.
It is thought that improving the bond durability between the feldspathic ceramics and the self-polymerizing resin through a limited procedure inside the mouth will lead to a strong temporary splint. The purpose of this study is to investigate the combined effect of two-liquid silane-phosphate primer and single-liquid sodium sulfite primer on the bonding between self-polymerizing resins and feldspathic ceramics, as well as the promotion of the polymerization behavior of two self-polymerizing resins at the bonding interface. In addition, differential scanning calorimetry (DSC) was conducted to analyze the kinetic polymerization behavior, particularly at the initial stage, of the self-polymerizing resins. The null hypotheses were set as follows: 1. Silane-phosphate primer has no effect on the shear bond strength and durability between the self-polymerizing resins and the feldspathic ceramics; 2. The use of single-liquid sodium sulfite primer after applied silane-phosphate primer has no effect on the shear bond strength and durability between the self-polymerizing resins and the feldspathic ceramics.

MATERIALS AND METHODS

Materials

One-hundred and thirty-two samples were prepared to compare the effects of two surface treatments on the shear strength of self-polymerizing resins bonded to feldspathic ceramics (Vitablocs Mark II, A3C I12, Vita Zahnfabrik, Bad Säckingen, Germany). Two types of surface treatment agents and self-polymerizing resins were assessed for the study. The materials applied are shown in Table 1.

Table 1  Materials assessed

| Material/Trade name                  | Manufacturer            | Lot  | Composition                                                                 |
|--------------------------------------|-------------------------|------|----------------------------------------------------------------------------|
| Adherend material                    |                         |      |                                                                            |
| Vitablocs Mark II                    | Vita Zahnfabrik         | 46680| SiO₂ 56–64%, Al₂O₃ 20–23%, Na₂O 6–9%, K₂O 6–8%, CaO 0.3–0.6%, TiO₂ 0.0–0.1% |
| A3C I12                              |                         | 48291|                                                                            |
| Primer                               |                         |      |                                                                            |
| Teeth Primer                         | Sun Medical             | ML1  | 4-META 10–30%, sodium sulfite 1–5%, acetone 20–40%, water                |
| Super-Bond PZ Primer A liquid        | Sun Medical             | MK1  | MMA, phosphate monomer, others                                            |
| Super-Bond PZ Primer B liquid        | Sun Medical             | MG1  | MMA, silane                                                               |
| Luting material                      |                         |      |                                                                            |
| Methyl methacrylate                 | Tokyo Chemical Industry | UQSHMSN | MMA 99.8%                                                                  |
| Super-Bond Monomer                  | Sun Medical             | LX1  | MMA, 4-META                                                               |
| Super-Bond Catalyst                 | Sun Medical             | LT1F | TBB, TBB-O, hydrocarbon                                                   |
| Super-Bond Ivory Opaque powder      | Sun Medical             | MM1  | PolyMMA, titanium oxide                                                   |

4-META, 4-methacryloxyethyl trimellitate anhydride; MMA, methyl methacrylate; TBB, tri-n-butylborane; TBB-O, partially oxidized tri-n-butylborane

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For the control group, no primers were applied to the plate surface.

For the PZ group, a PZ primer with equal amounts of liquids A and B was mixed and applied to the plate surface. After 10 s, the surface was dried in mild air.

For the PZ+TP group, a PZ primer was applied to the plate surface. After 10 s, the surface was dried in mild air. A TP primer was then applied to the surface.

Next, for all subgroups, a piece of double-sided adhesive tape with a circular hole (5 mm in diameter) was placed on the plate surface to define the bonding area. Subsequently, a stainless steel ring (SUS303) was placed on the tape and the self-polymerizing resin was filled into the hole using a brush. After 30 min, the bonded specimens were immersed in distilled water at a temperature of 37°C for 24 h. This state was set as the pre-thermocycle, and eleven specimens in each subgroup were subjected to a shear bond strength test at this stage. The other half of the specimens for each condition were thermocycled in water from 5°C to 55°C in temperature for 20,000 cycles using a thermocycling device (Thermal Shock Tester TTS-1 LM, Thomas Kagaku, Tokyo, Japan). The shear bond strength was then measured using a universal testing machine (Type 5567, Instron, Canton, MA, USA) at a crosshead speed of 0.5 mm/min. The experiment procedure is shown in Fig. 1.

**Failure mode analysis**

After the shear test, the fracture mode was determined using a microscope (Stemi DV4, Carl Zeiss, Jena, Germany) at an extended magnification of 16×. The failure mode analysis was classified into five categories as follows and described in Fig. 2:

A: adhesive failure including adherend-self-polymerizing resin interface separation

B: mixed failure including a combination of the adherend-self-polymerizing resin interface failure and cohesive failure in resin

C: cohesive failure inside the plate within the limits of the bonding area

D: cohesive failure inside the plate beyond scope of the bonding area

E: cohesive failure inside the plate without an adherend, namely, self-polymerizing resin interface separation

**Scanning electron microscopy observations**

Representative debonded ceramic surfaces were observed from six samples. The specimens were sputter coated with gold (Quick Coater SC-701, Sanyu Denchi, Tokyo, Japan) and then observed with a field-emission scanning electron microscope (SEM). The surfaces were analyzed to identify the bonding interface and cohesive failure areas.

**Fig. 1** Specimen preparation for shear bond test.

**Fig. 2** Classification of failure modes between the feldspathic ceramics and self-polymerizing resin. A: adhesive failure including adherend-self-polymerizing resin interface separation, B: mixed failure including a combination of the adherend-self-polymerizing resin interface failure and cohesive failure in resin, C: cohesive failure inside the plate within the limits of the bonding area, D: cohesive failure inside the plate beyond scope of the bonding area, E: cohesive failure inside the plate without an adherend, namely, self-polymerizing resin interface separation.
electron microscopy (SEM; ERA-8800FE, Elionix, Tokyo, Japan) with an accelerating voltage of 10 kV.

**DSC analysis**

A DSC analysis was conducted to analyze the effect of the application of the TP on the kinetic polymerization behavior of the MMA-TBB and 4-META/MMA-TBB resins using a differential scanning calorimeter (DSC-6100 Seiko Instruments, Tokyo, Japan). A sample was polymerized at 37°C isothermally in an aluminum pan (φ 5.2 mm, h 5.0 mm). For the sample preparation, 2.5 μL of TP was added to the sample in the pan, and 37.5 μL of the MMA or 4-META/MMA+PMMA slurry was then encapsulated, after which 2.5 μL of a polymerization initiator (TBB) was also added to the sample. The aluminum pan thus contained 42.5 μL of slurry. Because the MMA-TBB and 4-META/MMA-TBB resins polymerized rapidly with the mixing of the TBB, the measurement was started immediately after the TBB was dropped. The measurement was conducted under nitrogen gas for 60 min, and an empty aluminum pan was used as the reference material. The difference in polymerization activity was evaluated based on the shapes of the DSC curves.

**Table 2** Shear bond strength between feldspathic ceramics and MMA-TBB resin in MPa

| Groups   | Pre-thermocycles | Post-thermocycles | Post-/Pre-BS Ratio (%) | WRS |
|----------|------------------|-------------------|------------------------|-----|
|          | Median IQR       | Median IQR        |                        |     |
| Control  | 1.1a 1.2         | 0.0 0.0           | —                      | —   |
| PZ       | 28.2b 3.6        | 7.7c 1.2          | 27.3                   | S (p<0.0001) |
| PZ+TP    | 29.5b 2.3        | 13.0d 2.1         | 44.1                   | S (p<0.0001) |

n=11; PZ, Super-Bond PZ Primer with equal amounts of liquids A and B was mixed and applied the bonding surface.; PZ+TP, PZ primer was applied to the plate surface. After 10 s, the surface was dried in mild air. A Teeth Primer was then applied to the surface.; IQR, interquartile range; Post-/Pre-BS ratio, Post-/Pre-thermocycling bond strength ratio (%); WRS, where “S” indicates that the difference between the binary conditions of the pre- and post-thermocycling bond strength is significantly different (Wilcoxon rank sum test). Identical letters indicate that the values are not significantly different (Steel-Dwass multiple comparisons; p>0.05 and Wilcoxon rank sum test; p>0.05).

**Table 3** Shear bond strength between feldspathic ceramics and 4-META/MMA-TBB resin in MPa

| Groups   | Pre-thermocycles | Post-thermocycles | Post-/Pre-BS Ratio (%) | WRS |
|----------|------------------|-------------------|------------------------|-----|
|          | Median IQR       | Median IQR        |                        |     |
| Control  | 2.9A 0.7         | 0.2c 0.1          | 6.9                    | S (p<0.0001) |
| PZ       | 28.1b 4.9        | 5.4d 2.9          | 19.2                   | S (p<0.0001) |
| PZ+TP    | 27.3b 2.7        | 14.2e 6.2         | 52.0                   | S (p<0.0001) |

n=11; PZ, Super-Bond PZ Primer with equal amounts of liquids A and B was mixed and applied the bonding surface.; PZ+TP, PZ primer was applied to the plate surface. After 10 s, the surface was dried in mild air. A Teeth Primer was then applied to the surface.; IQR, interquartile range; Post-/Pre-BS ratio, Post-/Pre-thermocycling bond strength ratio (%); WRS, where “S” indicates that the difference between the binary conditions of the pre- and post-thermocycling bond strength is significantly different (Wilcoxon rank sum test). Identical letters indicate that the values are not significantly different (Steel-Dwass multiple comparisons; p>0.05).
Table 4 Failure mode analysis

| Groups       | Pre-thermocycles | Post-thermocycles |
|--------------|------------------|------------------|
|              | A    | B    | C    | D    | E    | A    | B    | C    | D    | E    |
| MMA-TBB      |      |      |      |      |      |      |      |      |      |      |
| Control      | 9    | 2    | 0    | 0    | 0    | 11   | 0    | 0    | 0    | 0    |
| PZ           | 0    | 0    | 0    | 0    | 11   | 0    | 1    | 10   | 0    | 0    |
| PZ+TP        | 0    | 0    | 0    | 0    | 11   | 0    | 0    | 0    | 11   | 0    |
| 4-META/MMA-TBB|     |      |      |      |      |      |      |      |      |      |
| Control      | 10   | 1    | 0    | 0    | 0    | 11   | 0    | 0    | 0    | 0    |
| PZ           | 0    | 0    | 0    | 1    | 10   | 0    | 3    | 8    | 0    | 0    |
| PZ+TP        | 0    | 0    | 0    | 1    | 10   | 0    | 0    | 0    | 11   | 0    |

thermocycle group, 21.40 for the 4-META/MMA-TBB resin of the pre-thermocycle group, and 28.67 for the post-thermocycle group. The $p$-value of the Kruskall-Wallis test was less than 0.001 for all groups.

Shear bond strength

The results of shear bond strength using MMA-TBB resin as the luting agent are summarized in Table 2. The median bond strengths of the pre-thermocycle group varied from 1.1 to 29.5 MPa. The PZ and PZ+TP groups
showed the highest shear bond strength (category b). A significant difference was seen between the control and the other groups (PZ: \( p<0.001 \) and PZ+TP: \( p=0.001 \)). The median bond strengths of the post-thermocycle group varied from 0.0 to 13.0 MPa. The PZ+TP group showed the highest shear bond strength (category d). A significant difference was seen between the PZ and PZ+TP groups (\( p<0.0001 \)).

The results of shear bond strength using 4-META/MMA-TBB resin as the luting agent are also summarized in Table 3. The median bond strengths of the pre-thermocycle group varied from 2.9 to 28.1 MPa. The PZ and PZ+TP groups showed the highest shear bond strength (category B). A significant difference was seen between the control and other groups (PZ: \( p<0.001 \) and PZ+TP: \( p<0.001 \)). The median bond strengths of the post-thermocycle group varied from 0.2 to 14.2 MPa. The PZ+TP group showed the highest shear bond strength (category E). A significant difference was seen among the control, PZ, and the PZ+TP groups (\( p<0.001 \)).

**Failure mode analysis**

The failure mode analyses are summarized in Table 4. The results of the failure mode analysis indicate that the high bond strength groups are in D or E mode, whereas the low bond strength groups are in A or B mode. Representative SEM images of debonded ceramic surfaces after thermocycles are shown in Figs. 3 and 4. Of specimens bonded with in the PZ group, the failure categories were B and C (Figs. 3a, c and 4a, c), whereas, for the PZ+TP group, the failure category was D (Figs. 3e and 4e).

**DSC analysis**

To further examine the mechanisms through which TP promotes the polymerization of the MMA-TBB and 4-META/MMA-TBB resins, a DSC analysis was conducted. The shapes of the DSC curves are shown in Fig. 5. In the groups in which polymerization initiator TBB were dropped into the MMA+PMMA and 4-META/MMA+PMMA slurry, the peak heat per unit of time occurred 20–60 min after the start of the measurement, whereas in the groups in which TP were added, the peaks appeared within 5 min after the start of the measurement.

**DISCUSSION**

According to the Kruskal-Wallis test for the shear bond strength of pre- and post-thermocycle group, the \( p \)-value was less than 0.001 for all groups. Hence, the first null hypothesis was rejected. Furthermore, the results of the Steel-Dwass multiple comparisons or Wilcoxon rank sum test indicated there was no significant difference between the PZ and PZ+TP groups in pre-thermocycle group, whereas a significant difference was shown between the PZ and PZ+TP groups in post-thermocycle group. Therefore, the second null hypothesis was partially rejected.

Kato et al. reported the bond durability between 4-META/MMA-TBB resin and feldspathic ceramics surface applied two-liquid silane-phosphate primer after polished with silicon-carbide (SiC) abrasive papers\(^8\). Its result indicated that the bond durability was greatly decreased by thermocycles. In this experiment, the bond durability of the group of PZ alone was also greatly decreased. However, the decrease of bond durability of PZ+TP group was suppressed. Therefore, the use of TP after applied PZ was suggested to be effective.

The TP primer used is composed of 4-META, sodium sulfite, acetone and water\(^{12-14}\). The purpose of this study was to investigate the combined effect of PZ primer, 4-META, and sodium sulfite on the bonding interface between self-polymerizing resin and feldspathic ceramics. Therefore, MMA-TBB resin which does not contain 4-META was selected as the luting agent. In addition, the effect of a commercial 4-META/MMA-TBB resin was also investigated. Since TP does not contain a silane compound, the application of TP alone was not tested in this experiment. Sodium sulfite and water were considered to be polymerization promoters for the MMA-TBB resin. It is thought that sodium sulfite, which is a reducing agent, improves the process of radical polymerization of a self-polymerizing resin\(^{16}\). Regarding the effect of water, Okamoto et al. reported that water assists in the formation and inhabitation of active species from partially oxidized tri-n-butylborane\(^{17}\). It
is therefore speculated that the use of TP improves the polymerization of MMA-TBB and 4-META/MMA-TBB resins.

The shear and tensile bond strength tests are common for bonding measurements between luting agents and adherend materials. Della Bona and van Noort compared the shear and tensile bond strengths between resin and feldspathic ceramics and concluded that the tensile bond strength test was a more effective method to assess the bond strength than the shear bond strength test18). In this experiment, however, MMA-TBB and 4-META/MMA-TBB resins cured products exhibit flexibility and stickiness. Therefore, the shear bond strength test was a more suitable testing method for the MMA-TBB resin than the tensile bond strength test.

DSC has been used to determine the working, setting, and polymerizing times of a self-polymerizing resin19,20). Several studies have been conducted on the kinetic polymerization behavior of a self-polymerizing resin using DSC19-21). Further, Imai et al. reported that the initiation of polymerization at the interface is important in the bonding of resin to the adherend material21). In the present study, a DSC measurement was conducted to analyze the effects of TP on the kinetic polymerization behavior of MMA-TBB and 4-META/MMA-TBB resins, and the peak heat per unit of time was shown to occur early through the addition of TP. These results indicate that a rapid initiation of polymerization of the TP and resins is beneficial for increasing the bond strength at the interface; therefore, it can be speculated that the PZ+TP group showed higher bond strengths than the PZ group. Furthermore, when comparing the shapes of the DSC curves of MMA-TBB resin and 4-META/MMA-TBB resin, the peak heat per unit of time occurred earlier in 4-META/MMA-TBB resin. It is indicated that exist of 4-META and the concentration of 4-META effects on the initial polymerization of self-polymerizing resin, and the rapid initiation of polymerization of resins has an effect on bond durability.

In clinical practice, saliva exists on the surface of ceramic restoration. Therefore, the influence of saliva contamination should be considered. Aboush reported that the use of phosphoric acid was the most beneficial as a surface cleaning method for porcelain22), and Aladaş et al. reported that the use of 0.5% sodium hypochlorite solution was beneficial for glass ceramics23). These reports suggest that surface cleaning can recover the bond strength between luting materials and ceramics. Hence, cleaning with phosphoric acid or sodium hypochlorite solution is considered desirable, although the influence of saliva was not considered in this experiment.

Application of TP was consequently shown to promote conversion during the initial polymerization of the self-polymerizing resin, and a firm bond was obtained at the bonding interface between the feldspathic ceramics and the self-polymerizing resin. As a result, it is proposed that the PZ+TP treatment be regarded as an effective treatment for a temporary splint used in teeth restoration.

CONCLUSION

When applying the MMA-TBB resin and 4-META/MMA-TBB resin for the temporary splint, the use of single-liquid sodium sulfite primer after the application of a silane-phosphate primer has an effect on bond durability as a surface treatment for teeth treated with feldspathic ceramic restorations.

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CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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