The effect of temporary sealing materials and cleaning protocols on the bond strength of resin cement applied to dentin using the resin-coating technique

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This study evaluated the effect of temporary sealing materials and cleaning protocols on the bond strength of resin cement applied to dentin using resin-coating technique. Scotchbond Universal Adhesive and Filtek Supreme Ultra Flowable were applied to bovine dentin. Forty-five specimens were divided into the following three groups according to the temporary sealing materials: Cav-: CAVITON EX, Vas-: COCOA BUTTER and FIT SEAL, and Sep-: Washable SEP and FIT SEAL. Each material was placed on resin-coated dentin. After 1-week water storage, one of the following three cleaning protocols was performed: -WA: washed with water, -BR: brushed with PRESSAGE, and -AF: cleaned with AIR-FLOW. Microtensile bond strength test and EDS analysis were conducted. Irrespective of the cleaning protocol used, Washable SEP demonstrated less residual debris on resin-coated dentin, resulting in high bond strength. Regardless of the temporary sealing material applied, AIR-FLOW demonstrated less residual debris, resulting in high bond strength.

Keywords: Resin coating, Resin cement, Dentin, μTBS

INTRODUCTION

In the resin-coating technique, the dentin and enamel surfaces that are exposed after cavity preparation are coated with a thin film of a coating material or a dentin bonding system combined with a flowable composite resin1. Several studies have demonstrated that the resin-coating technique protects the dentin and pulp2-4 through the formation of a hybrid layer, improving the resin cement–dentin bonding in an indirect restoration procedure5-9 and reducing the pain caused by external physical stimuli as it seals the dentinal tubules10-12.

The prepared cavity should be covered temporarily to provide positional stability and adequate occlusion and facilitate proper oral hygiene. However, the influence of remnants of provisional sealing material on the adhesion of final restoration is a significant concern. Nikaido et al.1,13 reported that resin-based filling materials may react with the resin-coating material and deteriorate the bond strength of the final luting cement. A water-setting material has been recommended for temporary sealing in the conventional procedure as it does not deteriorate the final bonding1,13. However, the water-setting material CAVITON EX (GC, Tokyo, Japan) could be easily dropped off from the cavity, especially in the early stage of hardening. Therefore, some clinicians rather prefer using resin-based temporary sealing material after applying Vaseline, e.g., COCOA BUTTER (GC), to the cavity to separate the resin-coated cavity from the resin-based temporary filling. Recently, the water-soluble separator Washable SEP (Sun Medical, Shiga, Japan) was launched for temporary sealing material. The manufacturer recommends using this material to resin-coated cavity as a separating agent from the resin-based temporary filling materials.

Fonseca et al.14 reported that the type of provisional cement and its removal method affected the adhesion of resin-bonded indirect restorations. The components of a temporary sealing material should ideally be completely removable from the surface. In this regard, a well-known procedure is the use of pumice with a rotational brush to remove the debris15. However, residual pumice can act as a contaminant for bonding16.

Some studies have reported the effectiveness of air polishing in removing plaque and stains17-19. Several types of air polishing powder are available. For instance, glycine (an amino acid with high water solubility) powders have lower hardness than sodium bicarbonate powders and cause less damage to the dentin surface and the gingival epithelium19. Furthermore, glycine powders were found to cause smaller changes in the dentin surface parameters than sodium bicarbonate powders20. Thus, the manufacturer recommends using AIR-FLOW powder SOFT (Electron Medical Systems, Nyon, Switzerland), which is composed of glycine powder, to clean the tooth before performing the restoration cementation procedure. However, there is limited evidence in the literature regarding the cleaning method of resin-coated dentin.

Therefore, this study was conducted to evaluate...
the effect of temporary sealing material and cleaning protocols of resin-coated dentin on the bond strength of resin cement. The null hypothesis of this study was that the selection of temporary sealing material and cleaning protocols of resin-coated dentin has no effect on the final bonding performance of resin cement.

**MATERIALS AND METHODS**

**Materials**
The materials used in this study are listed in Table 1. A light-emitting diode (LED) light-curing unit (VALO LED Curing Light; Ultradent Products, South Jordan,

### Table 1  Materials used in this study

| Materials          | Manufacturer          | Lot number | Main composition                                                                 |
|--------------------|-----------------------|------------|---------------------------------------------------------------------------------|
| **For resin-coating procedure** |                       |            |                                                                                 |
| Scotchbond Universal Adhesive | 3M ESPE, St. Paul, MN, USA | 70824A  | Bis-GMA, 2-hydroxyethyl methacrylate decamethylene, dimethacrylate, ethanol, water, silane-treated silica, 2-propenoic acid 2-ethyl, reaction products with 1,10-decanediol and phosphorus, oxide, copolymer of acrylic and itaconic acid, camphorquinone, dimethylaminobenzoate |
| Filtek Supreme Ultra Flowable (A2) | 3M ESPE            | N993427   | Bis-GMA, UDMA, TEGDMA, Bis-EMA, PEGDMA, silica, zirconia                     |
| **For cementing procedure** |                       |            |                                                                                 |
| RelyX Ultimate (Translucent) | 3M ESPE           | 4565965   | Base: methacrylate monomers, radiopaque silanated fillers initiator components, stabilizers, rheological additives catalyst paste: methacrylate monomers radiopaque alkaline fillers initiator components stabilizers, pigments, rheological additives fluorescence dye dark polymerize activator for Scotchbond Universal |
| CERASMART300 (Size 12, A2 LT) | GC, Tokyo, Japan   | 1708211   | Bis-MEPP, UDMA, DMA, silica, barium glass                                        |
| K-etchant GEL | Kuraray Noritake Dental, Tokyo, Japan | 5R0079 | 40% phosphoric acid, colloidal silica, water, dye                                |
| **For temporary sealing procedure** |                       |            |                                                                                 |
| CAVITON EX (White) | GC                   | 1901261   | Zinc oxide, plaster of Paris, vinyl acetate, ethanol                           |
| FIT SEAL | GC                   | Powder: 1807272 Liquid: 1810251 | PMMA, BPO, DBP, MMA, HEMA, BDDMA, DHEPT, ethanol                             |
| COCOA BUTTER | GC                   | 1808082   | Vaseline, cocoa butter                                                        |
| Washable SEP | Sun Medical, Shiga, Japan | ST1      | Ethanol, water-soluble polymer                                                   |
| **For cleaning procedure** |                       |            |                                                                                 |
| PRESSAGE | Shofu, Kyoto, Japan | 1118211   | Pumice, glycerin, CMC, paraben                                                |
| AIR-FLOW powder SOFT | Electron Medical Systems, Nyon, Switzerland | 111803 | Glycine-based particles (65 µm diameter)                                        |

Bis-GMA: bisphenol-A glycidylmethacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; Bis-EMA: ethoxylated bisphenol-a dimethacrylate; Bis-MEPP: 2,2-bis(4-methacycloxyethoxyphenyl) propane; DMA: dimethacrylate; PEGDMA: polyethylene glycol dimethacrylate; PMMA: polymethylmethacrylate; BPO: benzoyl peroxide; DBP: di-n-butyl phthalate; MMA: methyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; BDDMA: 1,3-butanediol dimethacrylate; DHEPT: N,N-di(2-hydroxyethyl)-p-toluidine; CMC: carboxymethyl cellulose.
UT, USA; high-power mode: 1,400 mW/cm²) was used in this study. Freshly extracted bovine incisors, stored frozen, were used as the bond strength test substrate. Forty-five teeth were used for microtensile bond strength test (5 teeth per group), and 30 teeth were used for a scanning electron microscope (SEM) observation and energy-dispersive X-ray spectroscopy (EDS) test (3 teeth per group). They were placed at room temperature and maintained moist to prevent dehydration 30 min before use. The labial surfaces were ground flat using a model trimmer (Y-230, Yoshida, Tokyo, Japan), until an area of approximately 60 mm² of dentin was exposed. To standardize the surface roughness and smear layer, the specimens were ground using a 600-grit SiC paper under running water for 60 s²¹-²³.

Resin coating
Scotchbond Universal Adhesive (3M ESPE, St. Paul, MN, USA) was applied to the dentin surface for 20 s, gently air thinned for 5 s, and then light cured for 10 s. Next, Filtek Supreme Ultra Flowable (A2, 3M ESPE) was placed as a thin layer on the cavity using a disposable applicator brush and light cured for 20 s²⁴. Then, they were wiped with an alcohol cotton swab to remove the low conversion layer on the resin-coated surface²⁵. The tooth specimens were randomly divided into nine groups (five teeth per group) after subjecting them to the three temporary sealing materials and the three cleaning protocols (Table 2).

| Group | Temporary sealing material | Cleaning protocol | Procedures |
|-------|---------------------------|-------------------|------------|
| Cav   | CAVITON EX                | Washed with distilled water | CAVITON EX was removed by hand excavator and then washed only with distilled water for 20 s. |
|       | BR                         | Brushed with PRESSAGE | CAVITON EX was removed by hand excavator, then brushed with PRESSAGE at 600 rpm for 30 s, and washed with distilled water for 5 s. |
|       | AF                         | AIR-FLOW           | CAVITON EX was removed by hand excavator, AIR-FLOW was performed at 10 mm distance for 10 s, and surface was washed with distilled water for 5 s. |
| Vas   | COCOA BUTTER, FIT SEAL    | Washed with distilled water | FIT SEAL was removed by hand excavator and then washed only with distilled water for 20 s. |
|       | BR                         | Brushed with PRESSAGE | FIT SEAL was removed by hand excavator, then brushed with PRESSAGE at 600 rpm for 30 s, and washed with distilled water for 5 s. |
|       | AF                         | AIR-FLOW           | FIT SEAL was removed by hand excavator, AIR-FLOW was performed at 10 mm distance for 10 s, and surface was washed with distilled water for 5 s. |
| Sep   | Washable SEP, FIT SEAL    | Washed with distilled water | FIT SEAL was removed by hand excavator and then washed only with distilled water for 20 s. |
|       | BR                         | Brushed with PRESSAGE | FIT SEAL was removed by hand excavator, then brushed with PRESSAGE at 600 rpm for 30 s, and washed with distilled water for 5 s. |
|       | AF                         | AIR-FLOW           | FIT SEAL was removed by hand excavator, AIR-FLOW was performed at 10 mm distance for 10 s, and surface was washed with distilled water for 5 s. |

rpm: rotations per minute.
The specimen aluminum holder, gold sputter coated, and sides were mounted using a carbon adhesion tape on the Bonferroni correction at a significance level of two-way analysis of variance (ANOVA) and sealing material and cleaning protocol were analyzed by Shapiro–Wilk test), the µTBS values of each temporary value was derived by dividing the imposed force at the minimum using a universal testing device (EZ-SX; Shimadzu, Kyoto, Japan). The microtensile bond strength (µTBS) measured using a digital caliper (Mitutoyo CD-15C, Kanagawa, Japan). In case pretest failures occurred during specimen preparation, the number of such failures was recorded, and the pretest failure was excluded from statistical calculations. As the distribution of data fitted the presumption of normality (based on the Shapiro–Wilk test), the µTBS values of each temporary sealing material and cleaning protocol were analyzed by two-way analysis of variance (ANOVA) and t-test with the Bonferroni correction at a significance level of α=0.05 in SPSS Statistics ver. 23.0 (IBM, Chicago, IL, USA).

After the µTBS test, the fractured specimens of both sides were mounted using a carbon adhesion tape on a specimen aluminum holder, gold sputter coated, and then observed under a SEM (JSM-IT100; JEOL, Tokyo, Japan) at 20.0 kV. The failure modes were categorized into the following five types: CoB, cohesive failure in the bond layer with >80% of total bonding area; CoC, cohesive failure in the cement layer with >80% of total bonding area; CoD, cohesive failure in the dentin with >80% of total bonding area; Mix, mixed failure; and Adh, adhesive failure with >80% of total bonding area. The specimens were then stored in a desiccator for 24 h, after which they were carbon coated and subjected to analysis of their microstructure morphology by SEM and EDS (JSM-IT100) at a working distance of 10 mm and a beam voltage of 20.0 kV.

**RESULTS**

**µTBS and failure modes**

Table 3 shows the results of the µTBS values and failure modes. The two-way ANOVA results indicated the presence of a significant interaction (p<0.01) and a significant effect for both the parameters’ temporary sealing material (p<0.01) and cleaning protocol (p<0.01).

Using CAVITON EX, the µTBS values of Cav-AF were significantly higher than those of Cav-WA (p<0.01) and Cav-BR (p<0.01). Using COCOA BUTTER and FIT SEAL, the µTBS values of Vas-WA were significantly lower than those of Vas-BR (p<0.01) and Vas-AF (p<0.01). In the case of Washable SEP and FIT SEAL, no significant differences were observed among the three removal method groups (p>0.05). For the “Washed by distilled water” group, Sep-WA had significantly higher µTBS values than Cav-WA (p<0.01) and Vas-WA (p<0.01). For the “Brushed with PRESSAGE” group, the µTBS values of Cav-BR were significantly lower than those of Vas-BR (p<0.01) and Sep-BR (p<0.01). Regarding the “AIR-FLOW” group, there were no significant differences among the three temporary sealing materials (p>0.05).

Representative SEM images of failure site are illustrated in Fig. 1. The majority of groups exhibited a predominant “cohesive failure in bond layer”. Meanwhile, “cohesive failure in resin cement layer or dentin” and “mix failure” appeared in the Vas-BR, Sep-BR, and Sep-AF groups. These groups demonstrated relatively high µTBS values among the tested groups. More than two pretest failures occurred in the Cav-WA, Cav-BR, and Vas-WA groups. These groups exhibited relatively low µTBS values of approximately 20 MPa, despite excluding the pretest failure specimens from the calculation.
Table 3 Results of microtensile bond strength

| Group | Temporary sealing material | Cleaning protocol | Mean±SD (MPa) | N/PTF | Failure mode |
|-------|---------------------------|-------------------|--------------|-------|--------------|
|       |                           |                   |              |       | CoB | CoC | CoD | Adh | Mix |
| Cav   | -WA                       |                   | 20.80±7.86 a, A | 18/2  | 12  | 0   | 0   | 0   | 6   |
|       | -BR                       |                   | 21.61±8.72 a, C | 16/4  | 14  | 0   | 0   | 0   | 2   |
|       | -AF                       |                   | 33.77±11.62 b, E | 20/0  | 12  | 0   | 0   | 1   | 7   |
| Vas   | -WA                       |                   | 20.79±8.38 c, A | 16/4  | 12  | 0   | 0   | 0   | 4   |
|       | -BR                       |                   | 41.39±12.07 d, D | 20/0  | 7   | 0   | 4   | 1   | 8   |
|       | -AF                       |                   | 36.66±15.87 d, E | 19/1  | 13  | 0   | 1   | 0   | 5   |
| Sep   | -WA                       |                   | 36.51±15.38 e, B | 19/1  | 14  | 0   | 0   | 0   | 5   |
|       | -BR                       |                   | 35.40±15.09 e, D | 20/0  | 5   | 2   | 1   | 2   | 10  |
|       | -AF                       |                   | 33.36±11.29 e, E | 20/0  | 7   | 0   | 0   | 0   | 13  |

Mean and standard deviation (MPa) of microtensile bond strength values (Number of tested specimens/pretest failures).

Within the same temporary sealing material group, the values with the same small letter are not significantly different ($p>0.05$).

Within the same cleaning protocol group, the values with the same capital letter are not significantly different ($p>0.05$).

Specimens were classified into five fracture modes as follows:

- **CoB**: cohesive failure in the bond layer with >80% of total bonding area;
- **CoC**: cohesive failure in the resin cement layer with >80% of total bonding area;
- **CoD**: cohesive failure in the dentin with >80% of total bonding area;
- **Adh**: adhesive failure with >80% of total bonding area;
- **Mix**: mixed failure.

N: number of specimens subjected to microtensile bond strength test; PTF: number of pretest failures; SD: standard deviation.

**SEM observation and EDS analysis**

Representative SEM images are depicted in Fig. 2. Some debris was observed in the Cav-WA, Cav-BR, and Vas-WA groups (Figs. 2b, c, and e). However, there was no debris in the other groups. Some tiny pits were observed in the AIR-FLOW-treated groups (Figs. 2d, g, and j). The EDS data of each element in mass percentage are presented in Table 4. All groups exhibited dominant proportions of carbon (C) and oxygen (O), whereas only 0.2% of silica (Si) was detected in the Cav-WA group, and >10% of Si was detected in the other groups. Zirconium (Zr) was detected in all groups, except in the Cav-WA group. Zinc (Zn) was detected in the Cav-WA and Ca-BR groups.
**DISCUSSION**

The mean µTBS values of the Cav-WA, Cav-BR, and Vas-WA groups were approximately 20 MPa, in spite of excluding the pretest failure specimens from the calculation. Moreover, a high number of pretest failures occurred in the Cav-WA, Cav-BR, and Vas-WA groups. These results were in agreement with the results of SEM and EDS analyses. In the Cav-WA, Cav-BR, and Vas-WA groups, debris was detected in the SEM findings (Figs. 2b, c, and e). In the EDS analysis, all groups were subjected to the resin-coating technique and application of carbon shadowing. Carbon shadowing contained C and O. Filtek Supreme Ultra Flowable, used as a resin-coating material, contain Si and Zr. Zn was the component of CAVITON EX. In the Cav-WA and Cav-BR groups, Zn was detected in the EDS analysis. For Vas-WA, it was obvious that COCOA BUTTER could not be removed by washing with distilled water. These findings indicated that the debris of CAVITON EX and COCOA BUTTER remained on the resin-coated dentin surface and interfered with the adhesion of resin cement in the Cav-WA, Cav-BR, and Vas-WA groups.

In contrast to our results, Nikaido et al.\(^26\) reported that a water-setting material increased the bond strength of resin cement, although the debris of water-setting materials remained on the resin-coated dentin. The probable reason for this inconsistency was the difference in the materials used. They used Cavit-G (3M ESPE) as a water-setting material, whereas we used CAVITON EX in our study. As the resin coating material, they used Protect Liner F (Kuraray Noritake Dental), whereas we used Filtek Supreme Ultra Flowable. The resin cements used in the previous study and our study

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**Table 4** Results of EDS analysis (mass %)

|       | C   | O   | Si  | Zr  | Ca  | Na  | P   | Zn  |
|-------|-----|-----|-----|-----|-----|-----|-----|-----|
| Baseline | 35.5 | 41.4 | 18.5 | 4.6  | 0   | 0.1 | 0   | 0   |
| Cav-WA   | 54.2 | 42.3 | 0.2  | 0    | 0.4 | 0.3 | 0.3 | 2.3 |
| Cav-BR   | 44.2 | 35.4 | 14.2 | 5.7  | 0.1 | 0.1 | 0.1 | 0.3 |
| Cav-AF   | 39.6 | 37   | 16.7 | 6.6  | 0   | 0.1 | 0   | 0   |
| Vas-WA   | 45.6 | 34.1 | 14.2 | 5.9  | 0.2 | 0.1 | 0   | 0   |
| Vas-BR   | 38.0 | 39.5 | 16.1 | 6.3  | 0   | 0.1 | 0   | 0   |
| Vas-AF   | 39.2 | 39.3 | 15.4 | 6    | 0   | 0.1 | 0   | 0   |
| Sep-WA   | 42.4 | 38.9 | 13.2 | 5.4  | 0   | 0.1 | 0   | 0   |
| Sep-BR   | 38.0 | 39.9 | 15.7 | 6.3  | 0   | 0.1 | 0   | 0   |
| Sep-AF   | 39.5 | 38.9 | 15.4 | 6.1  | 0   | 0.1 | 0   | 0   |

EDS: energy-dispersive X-ray spectroscopy; C: carbon; O: oxygen; Si: silicon; Zr: zirconium; Ca: calcium; Na: sodium; P: phosphorus; Zn: zinc.
were also different. Although the exact reason remains unclear, there might be some interaction among the water-setting material, the resin-coating material, and the resin cement.

A previous study on a cleaning protocol for contaminated dentin reported that some remaining particles of temporary cements were found in the group in which only the hand instrument was used\(^{27}\). To demonstrate the influence of the removal method, we performed three types of removal method (-WA, -BR, and -AF) following the hand instrument. Some studies have demonstrated that rotary instrumentation with pumice was an effective technique for mechanical cleansing protocol\(^{28,29}\). On the other hand, other studies have shown that the pumice method may not be very effective in every situation\(^{30,31}\). Our study results were consistent with the latter research because different temporary sealing materials exhibited different µTBS test results in the “-BR” group using rotary instrumentation with pumice.

The “-AF” group, in which AIR-FLOW was used, exhibited high µTBS values with all the temporary sealing materials compared with the other groups, and the number of pretreatment failures was also small. Furthermore, little debris and tiny pits were detected, which might have been caused because of AIR-FLOW particle collision as observed in the SEM images (Figs. 2d, g, and j). Previous studies using AIR-FLOW sodium bicarbonate powders concluded that the use of AIR-FLOW for surface preparation before restoration had no influence on the tensile bond strength to the enamel but adversely affected the bond strength to the dentin\(^{20,32}\). The TEM observations in those studies suggested that AIR-FLOW sodium bicarbonate powders could cause superficial maceration of the collagen fibers on the dentin surface\(^{33}\). Although glycine powders produced fewer defects\(^{34}\), the dentin surface should be protected by the resin-coating technique when using AIR-FLOW. SEM observation of this study showed that fillers of resin composite were observed clearly in the “-AF” groups (Figs. 2d, g, and j). Furthermore, Si of Baseline group was higher than that of other groups in the EDS results (Table 4). These features might show that base-resin of Filtek Supreme Ultra Flowable was slightly hollowed by AIR-FLOW. On the other hand, Zn was not detected in the Cav-AF group, whereas Zn was shown in the Cav-WA and Cav-BR groups. These findings indicated that AIR-FLOW contributed to the complete removal of temporary sealing materials, even CAVITON EX, from the resin-coated dentin surface.

The µTBS test revealed that the groups in which Washable SEP was used as a temporary sealing material showed similar bond strength regardless of the removable method. In the Washable SEP groups, no debris was observed in the SEM images (Figs. 2h–j) and Zn was not detected in the EDS analysis. The components of Washable SEP are hydrosoluble, e.g., ethanol and water-soluble polymers. Therefore, these findings may support the manufacturer’s recommendation of using this material in the tooth cavity with resinous materials as a separating agent from resin-based temporary filling materials.

Our results indicated that the selection of the temporary sealing material and the cleaning protocol had an influence on the final bonding performance of resin-coated dentin. The remaining debris of temporary sealing materials on the resin-coated dentin interfered with the adhesion of resin cement, resulting in lower bond strength. Therefore, the null hypothesis was rejected. It needs to be emphasized that these conclusions were derived from our study that dealt with a dentin bonding system in conjunction with a flowable resin composite. Although it has been reported that one-bottle coating materials resisted toothbrush abrasion\(^{35}\), the influence of air polishing on one-bottle coating materials is unknown. Further studies examining the use of one-bottle coating materials as resin coating are required.

CONCLUSION

Within the limitation of this study dealing with the resin-coating technique consisting of a dentin bonding system in conjunction with a flowable resin composite, both the selection of the temporary sealing material and the cleaning protocol influenced the bonding performance of resin cement applied to resin-coated dentin. Either using Washable SEP or applying AIR-FLOW resulted in less residual debris on the resin-coated surface, resulting in high bond strength.

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