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The Effect of Additional Silane Pre-Treatment on the Microtensile Bond Strength of Resin-Based Composite Post-and-Core Build-Up Material

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Abstract: The aim of this study was to evaluate the effect of additional silane treatment on the immediate and aged microtensile bond strength (μTBS) between resin-based composite (RBC) post-and-core build-up material and an RBC CAD/CAM block. Twelve sample blocks (12 mm × 14 mm × 9 mm) were prepared using RBC post-and-core build-up material and were divided into six groups: Kerr Silane Primer (Sil) + OptiBond eXTRa Universal adhesive (EXA); OptiBond eXTRa Universal Primer (EXP) + EXA; Sil + OptiBond Universal (OBU); OBU; Sil + OptiBond Solo Plus (OSP); and OSP. Each treated sample was luted to a CAD/CAM block using an NX3 Nexus Third-Generation instrument. After storage in 37 °C water for 1 week, microspecimens were fabricated, and μTBS was tested immediately or after further immersion in water at 37 °C for 6 months. The failure mode of each specimen was determined using stereomicroscopy and scanning electron microscopy. For the immediate groups, no significant difference in μTBS was found between specimens with or without silane treatment for each adhesive ($p > 0.05$). For the aged groups, silane treatment significantly decreased μTBS for OptiBond eXTRa Universal ($p < 0.05$). Our findings indicate that additional silane treatment prior to the application of adhesive did not enhance μTBS.

Keywords: silane; CAD/CAM; post-and-core build-up; microtensile bond strength; universal adhesive

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1. Introduction

Crown restorations using resin-based composite (RBC) CAD/CAM blocks require both high adhesion to the tooth substance by luting cement and high adhesion to the RBC block because the integration of the abutment tooth and CAD/CAM restoration by adhesion affects the subsequent fracture resistance [1]. The bond strength of resin-based luting material to tooth substance has been investigated in previous studies [2,3]. Although the bond strength varies with each product [4], almost all commercially available products have proven to be clinically acceptable [5–7]. Many studies have also been conducted on the optimal protocol for bonding these materials to RBC CAD/CAM blocks [8]. Some studies reported that alumina sandblasting followed by silane treatment of the bonded surface of the block is optimal [9–12], while one study found that silane treatment was not effective for adhered surfaces etched with hydrofluoric acid [13].

Endodontically treated teeth often have extensive loss of coronal tooth structure. Therefore, many cases require post-and-core build-up placement prior to coronal restoration. This method has been applied to endodontically treated teeth for more than three centuries [14]. Gold alloys and platinum gold alloys are generally used for cast post-and-core materials [15,16]. In Japan, cast post-and-core build-ups using silver-based alloys have been used for a long time because they are covered by national health insurance [17]. Recent studies have reported that cast post-and-core materials have good clinical outcomes similar to those of fiberglass posts [18]. The main disadvantage of these metal materials is their poor esthetics; while restorations using porcelain fused to metal cast are not affected [19], all-ceramic restorations are affected by the color tone of the underlying post-and-core material, necessitating the use of color masking [20,21].

Recently, post-and-cores with RBC reinforced with a fiber post have been widely adopted [22,23]. This method has high fracture resistance similar to that of cast post-and-core materials [24]. Additionally, the color of RBC is similar to that of the remaining coronal dentin, so the color mismatch is unlikely to occur even with all-ceramic restorations [25].

Unfortunately, there is no consensus on the optimal surface treatment for abutment tooth built-up with fiber-reinforced RBC post-and-core materials prior to luting, because few studies have investigated the bond strength of luting cement to the RBC build-up material [26–28]. Additionally, these previous studies only examined the effects of the difference in the luting material, cleaning protocol, and surface treatment on bond strength. Therefore, there is no previous study examining the effect of silanization. It is more difficult to distinguish the boundary between the build-up material and the remaining coronal dentin than the boundary between a cast post-and-core build-up and the remaining coronal dentin. It is therefore possible that the adhesion of the luting cement to the remaining coronal dentin might be inhibited due to contamination by silane. It is desirable to avoid applying different pre-treatment protocols to both materials.

This study was therefore conducted to evaluate the effect of additional silane treatment on the immediate and aged microtensile bond strength (μTBS) between the RBC post-and-core build-up material and the RBC CAD/CAM block. The null hypothesis was that additional silane treatment on the surface of the RBC post-and-core material does not improve the immediate and aged μTBS.

2. Materials and Methods

2.1. Materials Used in This Study

The materials used in this study and the respective manufacturers, compositions, and batch numbers are described in Table 1. Estecore (Universal shade, Tokuyama Dental, Kamisu, Japan) was used as the RBC post-and-core build-up material. KZR-CAD HR3 GAMMATHETA (Shade A3, YAMAKIN, Konan, Japan) was used as the RBC CAD/CAM block. Multi-Etchant (YAMAKIN) was used for surface treatment of the CAD/CAM block, and NX3 Nexus Third-Generation Automix Dual-Cure Syringe (Kerr, Orange, CA, USA) was used for the RBC luting cement. The adhesive systems used in this study were OptiBond eXTRa Universal (Kerr), a 2-step self-etch adhesive; OptiBond Universal (Kerr), an all-in-one adhesive; and OptiBond Solo Plus, a 2-step etch-and-rinse adhesive. Kerr Silane Primer (Kerr) was used as the silane coupling agent.

Table 1. Composition and instructions for use of materials used in the study.

Materials (Manufacturer)	Batch No.	Main Components *	Procedure
[post-and-core material]			
Estecore (Tokuyama Dental, Kamisu, Ibaraki, Japan)	279041	Paste A: silica zirconia filler, Bis-GMA, TEGDMA, Bis-MPEPP Paste B: silica zirconia filler, Bis-GMA, TEGDMA, Bis-MPEPP, peroxides, camphorquinone, radical amplifiers.	-

Table 1. *Cont.*

Materials (Manufacturer)	Batch No.	Main Components *	Procedure
[Resin-based composite CAD/CAM block]			
KZR-CAD HR3 GAMMATHETA (YAMAKIN, Konan, Kochi, Japan)	01052117	UDMA, DEGDMA, SiO_2-Al_2O_3-ZrO_2, SiO_2, filler mass (75wt%)	-
[Silane coupling agent]			
Kerr Silane Primer (Kerr, Orange, CA, USA)	7970022	Ethyl alcohol, organosilane ester	Apply and dry with a compressed air syringe (not light-cured).
[Adhesive]			
OptiBond eXTRa Universal (Kerr, Orange, CA, USA)	7941589	Primer: GPDM, HEMA, acetone, ethyl alcohol, water, initiators	Apply the primer with a brushing motion for 20 s, gently air-dry for 5 s.
	7945265	Adhesive: ethyl alcohol, alkyl dimethacrylate resins, barium aluminoborosilicate glass, fumed silica (silicon dioxide), and sodium hexafluorosilicate	Apply adhesive to Estecore and KZR-CAD HR3 surface for 15 s. Air-thin with gentle air first, then apply strong air for at least 5 s to avoid pulling; do not light-cure.
OptiBond Universal (Kerr, Orange, CA, USA)	7982104	GPDM, HEMA, dimethacrylate, acetone, ethanol, glycerol	Apply adhesive to the Estecore surface for 20 s. Air-thin with gentle air first, and then apply mild air for at least 5 s to avoid pulling; do not light-cure.
OptiBond Solo Plus (Kerr, Orange, CA, USA)	7906160	Gel Etchant: 37.5% phosphoric acid, silica thickener. Primer/adhesive: Bis-GMA, GDM, HEMA, GPDM, ethanol, aluminum borosilicate glass, fumed silica, sodium hexafluorosilicate, photoinitiator	After applying Gel Etchant for 15 s, rinse for 15 s, and gently air-dry. Bond for 15 s and gently air-dry for 10 s; do not light-cure.
[Resin-based composite luting agent]			
NX3 Nexus Third-Generation Universal Adhesive Resin Cement (Kerr, Orange, CA, USA)	7927410	Methacrylate ester monomers, inert mineral fillers, activators, stabilizers, and a radiopaque agent	After completing the bonding protocol on the EC surface, dispense cement with an automix syringe onto the KZR3 surface. Light-cure on the four proximal sides each for 20 s.
[Etching agent]			
Multi-Etchant (YAMAKIN, Konan, Kochi, Japan)	01101929	Purified water, M-TEG-P, thickener, dyes	Apply for 15 s with rubbing motion and let stand for 10 s. Rinse thoroughly with water and dry with oil-free compressed air.

* Bis-EMA: ethoxylated bisphenol-A-dimethacrylate, Bis-GMA: bisphenol-A-diglycidyl methacrylate, Bis-MPEPP: 2,2-bis(4-methyacryloxyethoxyphenyl) propane, DEGDMA: diethyleneglycol dimethacrylate, UDMA: urethane dimethacrylate GDM: glycerol dimethacrylate, GPDM: glycerol phosphate dimethacrylate, HEMA: 2-hydroxyethyl methacrylate, M-TEG-P: methacryloyloxydecyl tetraethyleneglycol phosphate, TEGDMA: triethyleneglycol dimethacrylate, UDMA: urethane dimethacrylate.

2.2. Preparation of Specimen Blocks from RBC Post-and-Core Build-Up Material

A schematic illustration of the specimen preparation is shown in Figure 1. Pastes A and B of Estecore were mixed with a mixing tip, pressed into silicone molds using a glass slide (Matsunami Glass Industries, Kishiwada, Japan), and photo-polymerized using a light-emitting diode (LED) light-curing unit (Demi Plus, Kerr) to prepare 12 sample blocks (12 mm × 14 mm × 9 mm). Estecore was carefully condensed in order to avoid void

entrapment. One side of the block surface (12 mm × 14 mm) was abraded using 600-grit SiC paper (Fuji Star Waterproof Abrasive Paper Sheet, Sankyo Rikagaku, Okegawa, Japan) to prepare a uniform and flat surface.

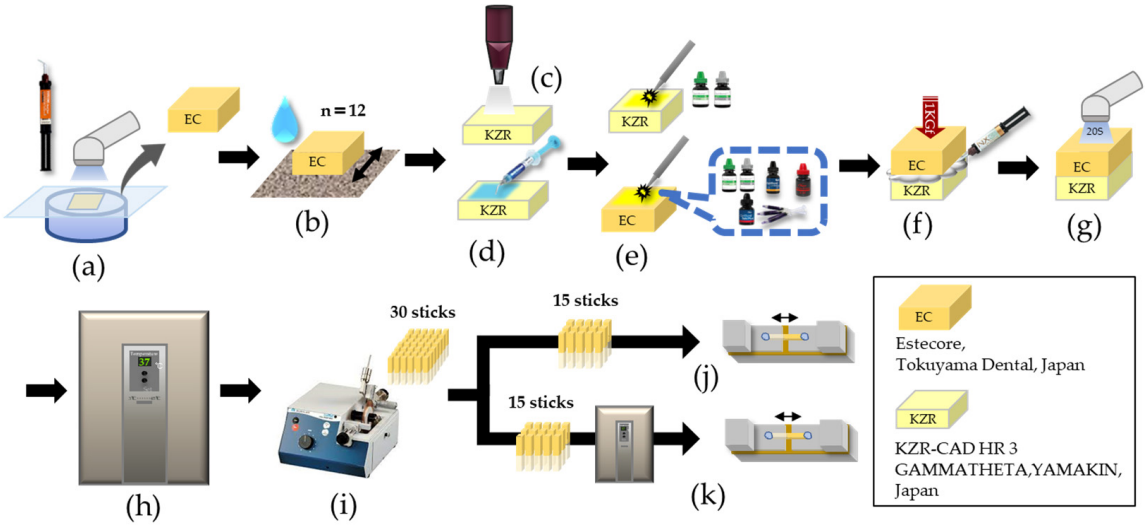


Figure 1. Schematic illustration of the experimental setup for microtensile bond strength (μTBS) testing: (**a**) resin-based composite for core build-up (Estecore) was packed into the silicone mold; (**b**) grinding of the adhered surface with #600 SiC paper; (**c**) alumina airborne particle abrasion with 50 μm particles; (**d**) pre-treatment using Multi-Etchant; (**e**) application of primer/adhesive; (**f**) luting of resin-based composite CAD/CAM block; (**g**) light-curing for 80 s and waiting for 30 min; (**h**) storage of specimens in water at 37 °C for 1 week; (**i**) cutting into 1 mm^2 stick-shaped microspecimens; (**j**) μTBS testing immediately after cutting; (**k**) μTBS testing after further storage in water at 37 °C for 6 months.

2.3. Pre-Treatment of Post-and-Core RBC Blocks

The abraded surfaces were etched with Gel Etchant (Kerr) and randomly assigned into six experimental groups (n = 2).

Group 1 (Sil+EXA): Silane Primer was applied and dried with compressed air, and OptiBond eXTRa Universal Adhesive was applied.

Group 2 (EXP+EXA): OptiBond eXTRa Universal Primer was applied and dried with compressed air, and OptiBond eXTRa Universal Adhesive was applied.

Group 3 (Sil+OBU): Silane Primer was applied and dried with compressed air, and OptiBond Universal was applied.

Group 4 (OBU): only OptiBond Universal was applied (without silane treatment).

Group 5 (Sil+OSP): after conditioning with Gel Etchant, the conditioned surface was kept moist, Silane Primer was applied and dried using compressed air, and OptiBond Solo Plus was applied.

Group 6 (OSP): after conditioning with Gel Etchant, the conditioned surface was kept moist, and OptiBond Solo Plus was applied.

These surface slabs were stored in the dark until immediately before use.

2.4. Preparation of CAD/CAM Blocks

RBC CAD/CAM blocks (KZR-CAD Block 3 GAMMA THETA, Shade A3, 12 mm × 14 mm × 18 mm, YAMAKIN) were cut into two halves (12 mm × 14 mm × 9 mm) using a low-speed water-cooled diamond saw (Isomet, Buehler) to yield 12 blocks. The cut surface was abraded using 600-grit SiC paper (Fuji Star Waterproof Abrasive Paper Sheet) to prepare a uniform and flat surface. The abraded surfaces were then subjected to alumina airborne particle abrasion with 50 μm particles (J. Morita, Tokyo, Japan) using an airborne particle abrader (Jet Blast II, J. Morita) for 20 s at a pressure of 0.2 MPa and a distance of 10 mm from the target surface. The blocks were pre-treated using Multi-Etchant (YAMAKIN) for

15 s, thoroughly rinsed with water spray, and dried with compressed air. Kerr Silane Primer was applied to the surface and thinned with compressed air. Thereafter, OptiBond eXTRa Universal Adhesive was further applied and thinned using compressed air, and the blocks were stored in the dark until immediately before use.

2.5. Bonding Procedures

The pre-treated CAD/CAM blocks were luted to the pre-treated RBC post-and-core build-up material surfaces using NX3 Nexus Third-Generation instrument under a constant seating force of 1 kgf [29]. They were immediately light-cured from the four proximal sides of all specimens for 20 s each (for a total of 80 s per specimen), and constant pressure was applied for 30 min. The adhesive on the post-and-core RBC surface and the luting cement was polymerized with an LED curing unit with a light intensity of 1400 mW/cm^2 (Demi Plus, Kerr). The light intensity output was regularly checked with a radiometer (Bluephase meter II; Ivoclar Vivadent, Schaan, Liechtenstein) throughout the entire luting procedure [30].

After storage in water at 37 °C for 1 week, specimens were serially sectioned perpendicularly to the adhered surface using a low-speed water-cooled diamond saw (Isomet) to obtain rectangular 1 mm × 1 mm stick-shaped microspecimens (Figure 2). Thirty of the obtained microspecimens were selected from the center of each specimen and visually examined. The microspecimens with obvious defects, such as voids and gaps within the built-up composite, were eliminated before bond testing. Half of the microspecimens were attached to a microtensile jig using cyanoacrylate glue (Model Repair II Blue, Morita Dental Products, Ohtawara, Japan) and subjected to a tensile load using a microtensile tester (Bisco, Schaumburg, IL, USA) at 1.0 mm/min crosshead speed until failure (immediate group). The other half of the sticks in each group were further immersed in water at 37 °C for 6 months and tested for μTBS (aged group). The width and thickness of each specimen were measured to the nearest 0.01 mm using a digital caliper (CD-15 CPX, Mitutoyo, Kawasaki, Japan) to determine the microtensile bond strength (μTBS, MPa) by dividing the recorded force (N) at the time of fracture by the bond area (mm^2). If a specimen failed before proper testing, a bond strength of 0 MPa was used for statistical analyses [31–33]. The actual number of pre-testing failures was also explicitly noted.

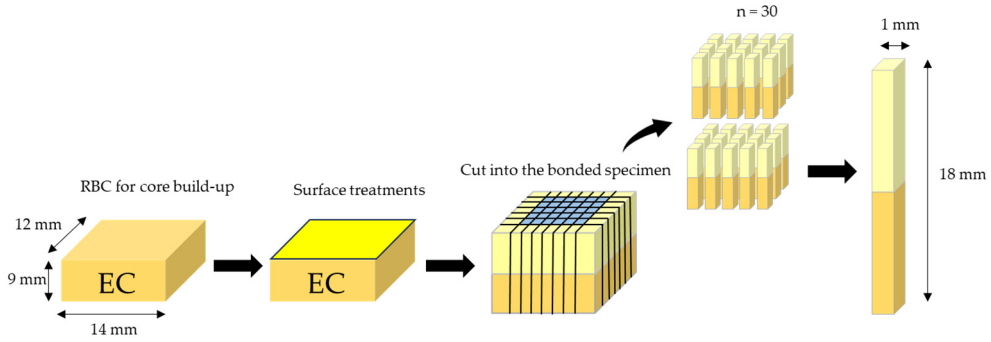


Figure 2. Schematic illustration of the preparation of microspecimens for microtensile bond strength (μTBS) testing.

2.6. Statistical Analysis

The adequate minimum sample size for μTBS testing was calculated as follows:

$$n = 2\{(Z_{\alpha/2} + Z_{\beta})^2 \times \sigma^2\}/\Delta^2, \quad (1)$$

where n is the minimum sample size, α is the probability of falsely rejecting a true null hypothesis (α = 0.05, $Z_{\alpha/2}$ = 1.96), β is the probability of failing to reject a false null hypothesis (β = 0.20, Z_{β} = 0.84), σ is the standard deviation, and Δ is the difference among groups. According to the calculation, at least 23 specimens were needed in each group.

Prior to hypothesis testing, the Shapiro–Wilk test was used to determine whether the bond strength data had a normal distribution. It was decided to use a three-way analysis of variance (ANOVA) with the three adhesive systems, silanization, and storage periods as primary parameters for analysis. According to the assumption of homogeneity of variance, Tukey's HSD test (adhesive) and the unpaired *t*-test (silanization and storage period) were used for the storage period and μTBS values, respectively. The critical value was 0.05. All statistical analyses were carried out using SPSS statistical software (IBM SPSS 18; SPSS Inc., Chicago, IL, USA).

2.7. Analysis of Failure Modes

The failure mode was determined at a 50× magnification with a stereomicroscope. The failure modes were categorized into the following four groups:

- Type A: complete adhesive failure between the post-and-core RBC and CAD/CAM RBC block;
- Type C-1: cohesive failure in the post-and-core RBC;
- Type C-2: cohesive failure in the CAD/CAM RBC block;
- Type M: a mixture of adhesive and cohesive failures.

After stereomicroscopic observation, the representative specimens in each group were selected for scanning electron microscopy (SEM). After being mounted on stubs, the specimens were Au-Pd-coated using a cooled sputter coater (MSP-20-UM Automatic Magnetron Sputter, Vacuum Device, Mito, Japan). The coated specimens were examined using SEM (SU6600, Hitachi, Tokyo, Japan) at 15 kV.

3. Results

3.1. μTBS

The results of μTBS for each group are shown in Table 2. Three-way ANOVA showed that the factors of 'adhesive' and 'storage period' both had a significant influence ($F = 38.612$, $p < 0.001$; $F = 1943.799$, $p < 0.001$, respectively). In contrast, 'silanization' had no significant effect ($F = 3.277$, $p = 0.071$). Significant interaction could not be detected between the factors of 'adhesive' and 'storage period' ($F = 1.180$, $p = 0.154$) or between the factors of 'silanization' and 'storage period' ($F = 0.078$, $p = 0.780$). However, there was a significant interaction between the factors of 'adhesive' and 'silanization' ($F = 11.075$, $p < 0.001$). The interaction among the three factors was not significant ($F = 1.088$, $p < 0.338$).

Table 2. Results of the mean microtensile bond strength (μTBS, MPa) ** and standard deviations (S.D.) of each group.

Storage Group	OptiBond eXTRa Universal		OptiBond Universal		OptiBond Solo Plus	
	with Silane	w/o Silane (with Primer)	with Silane	w/o Silane	with Silane	w/o Silane
Immediate (PTF/*n*) *	58.01 ± 4.68 ^{abc,A} 0/30	61.17 ± 4.46 ^{a,A} 0/30	54.70 ± 11.51 ^{bc,A} 0/30	52.60 ± 6.38 ^{c,A} 0/28	59.31 ± 9.64 ^{ab,A} 0/30	61.41 ± 3.06 ^{a,A} 0/30
Aged (PTF/*n*) *	27.87 ± 5.89 ^{b,B} 0/29	33.90 ± 5.04 ^{a,B} 0/30	25.27 ± 7.26 ^{bc,B} 1/30	21.06 ± 6.03 ^{c,B} 1/30	27.15 ± 4.17 ^{b,B} 0/30	29.64 ± 4.04 ^{ab,B} 0/28

* *n*, total number of bonded specimens; PTF, pre-testing failure; ** the same lowercase superscript letters within each column indicate the absence of statistically significant difference ($p > 0.05$); the same uppercase superscript letters within each row indicate the absence of statistically significant difference ($p > 0.05$).

For immediate groups, no significant difference was found between the presence and absence of silane treatment for each adhesive ($p > 0.05$). For aged groups, we also found no significant difference between the presence and absence of silane treatment for OptiBond Universal and OptiBond Solo Plus ($p > 0.05$). Only in the case of OptiBond eXTRa Universal was the μTBS of Group 2 (pre-treated with OptiBond eXTRa Universal Primer but without the application of Silane Primer) significantly higher than that of Group 1 (pre-treated with Silane Primer but without the application of OptiBond eXTRa Universal Primer).

3.2. Failure Modes

The distributions of the failure modes according to immediate and aged groups are summarized in Figure 3.

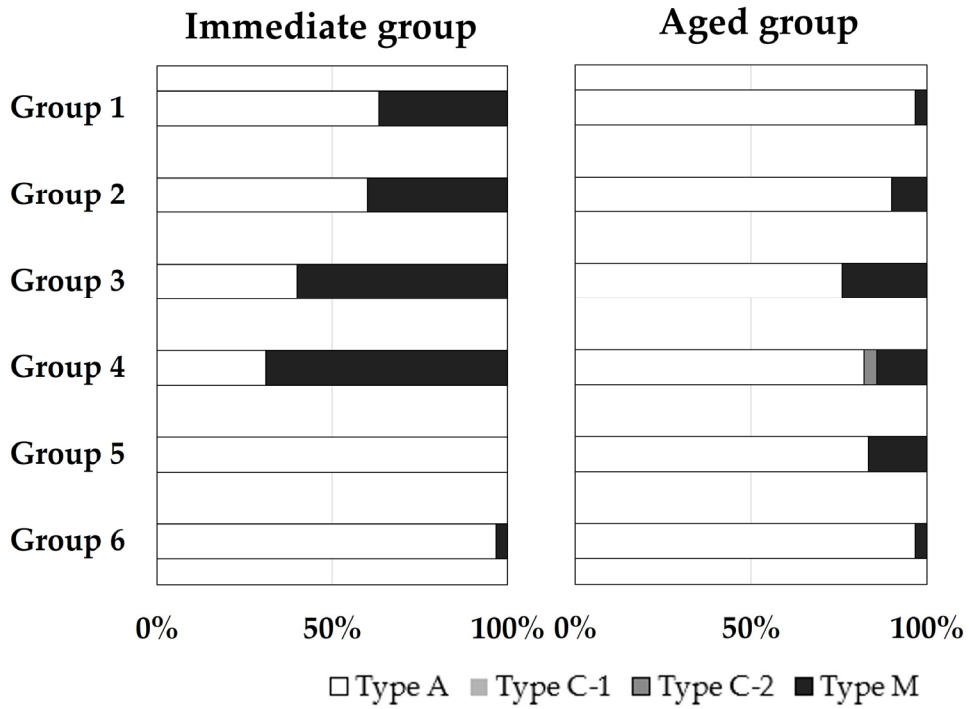


Figure 3. Fracture mode distribution in percentage (%) for immediate and aged groups: Type A: complete adhesive failure between the post-and-core RBC and CAD/CAM RBC block, Type C-1: cohesive failure in the post-and-core RBC, Type C-2: cohesive failure in the CAD/CAM RBC block, Type M: mixture of adhesive and cohesive failures.

For the immediate groups, OptiBond eXTRa Universal groups (Groups 1 and 2) mainly failed at the adhesive interface (63.3% and 60.0%, respectively) rather than exhibiting a mixed failure (36.7% and 40.0%, respectively), and specimens remained intact. OptiBond Solo Plus groups (Groups 5 and 6) mainly failed at the adhesive interface (100% and 96.7%, respectively). In contrast, OptiBond Universal groups (Groups 3 and 4) displayed mainly mixed failure (60.0% and 69.0%, respectively) rather than failure at the adhesive interface (40.0% and 31.0%, respectively).

For aged groups, all six groups mainly failed at the adhesive interface (96.7%, 90.0%, 75.9%, 82.1%, 83.3%, and 96.6%, respectively) rather than a mixed failure (3.3%, 10.0%, 24.1%, 14.3%, 16.7%, and 3.3%, respectively). Only for one specimen in Group 4 did we observe a cohesive failure in the CAD/CAM RBC block.

Representative SEM images of the fractured surfaces after μTBS testing of immediate groups are shown in Figure 4. Figure 4A,B are examples of Type A. In Figure 4A, fine undulations are observed on almost the entire fracture surface, while in Figure 4B, scratches formed by SiC paper can be seen on part of the fracture surface. Figure 4C shows the fracture surface on the RBC CAD/CAM block side. Most of the fractured surface has fine undulations similar to those in Figure 4A. However, an obvious scratch is observed in the upper right part of the fracture surface, and a layer of what appears to be adhesive is observed between fine undulations and the scratch. In Figure 4D, scratches are observed on more than half of the fracture surface, and a characteristic image of a broken CAD/CAM block can be seen in the lower right part of the fracture surface.

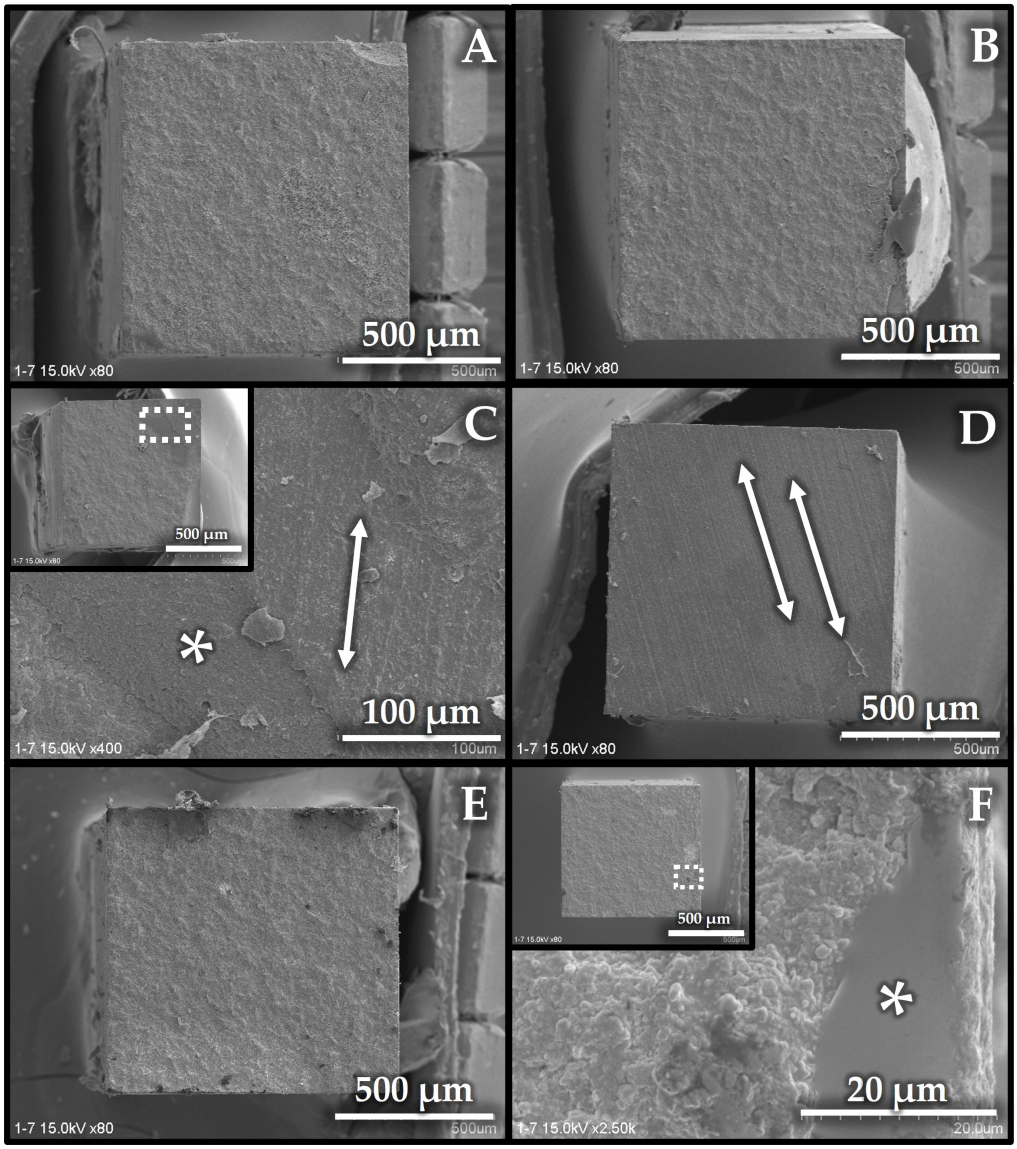


Figure 4. Representative scanning electron microscope images of the fractured surfaces after microtensile bond strength (µTBS) testing in the immediate groups: (**A**) OptiBond eXTRa Universal with silane, build-up material side. Fine undulations can be seen; (**B**) OptiBond eXTRa Universal with OptiBond eXTRa Universal Primer (without silane), build-up material side. Fine undulations are evident; (**C**) OptiBond Universal with silane, CAD/CAM-block side. Obvious scratches (double-sided arrows) and failure within the cement layer (asterisk) are observed; (**D**) OptiBond Universal without silane, build-up material side. Obvious scratches (double-sided arrows) can be seen; (**E**) OptiBond Solo Plus with silane, build-up material side. Fine undulations are evident; (**F**) OptiBond Solo Plus without silane, build-up material side. Fine undulations and failure within the cement layer (asterisk) can be seen.

Representative SEM images of the fractured surfaces after µTBS of aged groups are shown in Figure 5. In Figure 5A,B,D,F, fine undulations can be seen on most of the fracture surfaces. In Figure 5C, scratches formed by SiC paper are evident on almost the entire fracture surface. In Figure 5E, scratches can be seen on the upper left and lower right sides of the fracture surface.

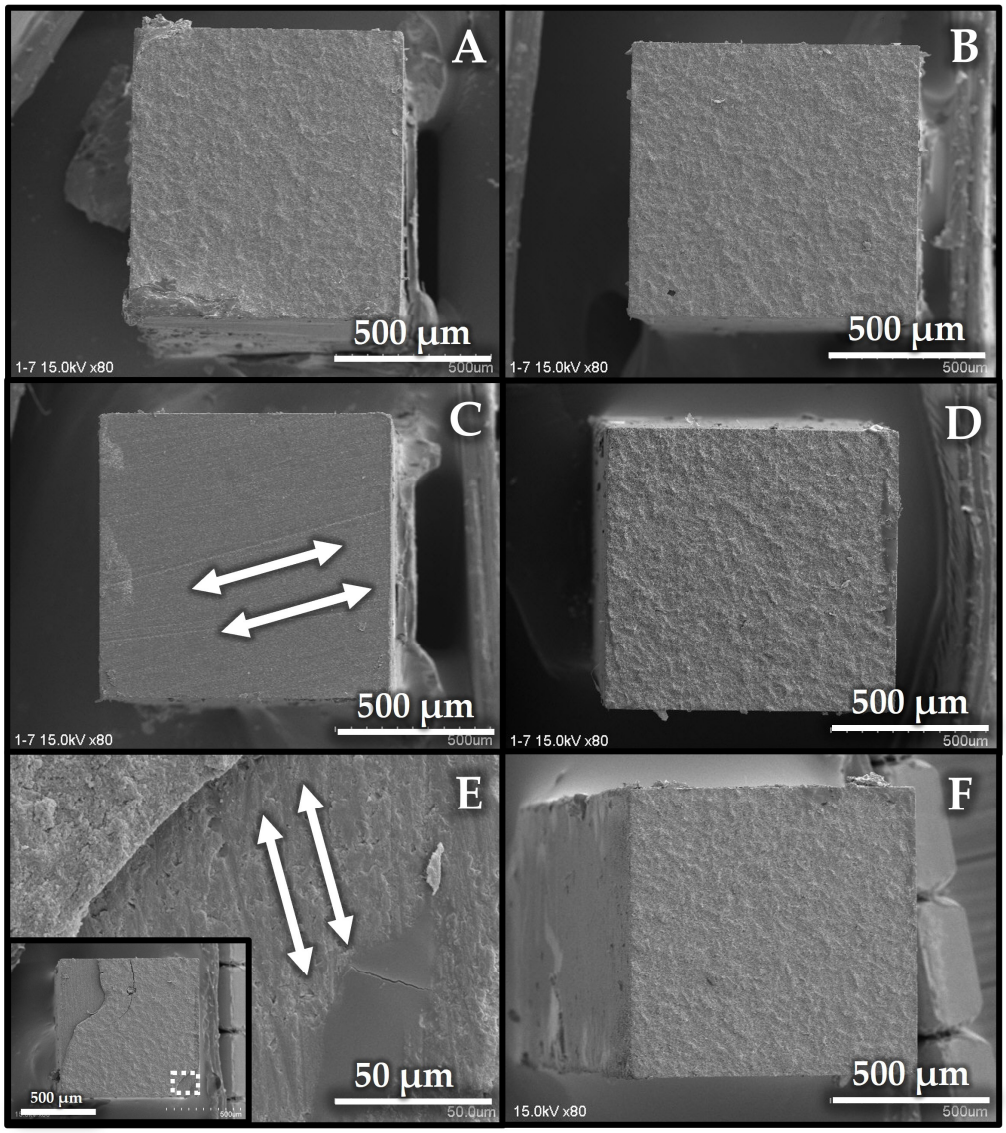


Figure 5. Representative scanning electron microscope images of the fractured surfaces after microtensile bond strength (µTBS) in the aged groups: (**A**) OptiBond eXTRa Universal with silane, build-up material side. Fine undulations can be seen; (**B**) OptiBond eXTRa Universal with OptiBond eXTRa Universal Primer (without silane), build-up material side. Fine undulations are evident; (**C**) OptiBond Universal with silane, build-up material side. Obvious scratches (double-sided arrows) are visible; (**D**) OptiBond Universal without silane, build-up material side. Fine undulations are evident; (**E**) OptiBond Solo Plus with silane, build-up material side. Fine undulations are visible, and obvious scratches can be seen (double-sided arrows); (**F**) OptiBond Solo Plus without silane, build-up material side. Fine undulations are evident.

4. Discussion

The purpose of this study was to investigate the effect of additional silane treatment applied to RBC post-and-core build-up material on the immediate and aged µTBS of RBC CAD/CAM blocks. The results revealed that no benefit was evident from additional silane treatment for OptiBond eXTRa Universal, OptiBond Universal, or OptiBond Solo Plus. Additional silane treatment prior to the application of OptiBond eXTRa Universal reduced the microtensile bond strength in the aged group. Therefore, the null hypothesis could not be rejected regardless of the duration of the immersion in water.

NX3 is an RBC luting material and does not contain adhesive monomers or amines. Therefore, it is reported to have relatively less water absorption [34] and less long-term

color change [35] compared with common adhesive resin cements and self-adhesive resin cements. However, its application requires a combination with an adhesive system [36], and the OptiBond series is the recommended adhesive system. The OptiBond series includes the three-step FL, the two-step self-etching eXTRa Universal, the two-step etch-and-rinse Solo Plus, and the one-step Universal. Unfortunately, OptiBond FL is not distributed in Japan and is difficult to obtain. This study therefore tested and compared the other three products.

OptiBond eXTRa Universal, classified as a two-step self-etch adhesive, is the successor to OptiBond XTR. OptiBond XTR has been reported to have high adhesive strength to both enamel and dentin, similar to Clearfil SE Bond (Kuraray Noritake Dental) [37–39], one of the gold standard dental adhesives [40]. Unlike Clearfil SE Bond, OptiBond XTR contains ethanol as a solvent, which causes insufficient polymerization due to residual ethanol if thorough air-drying is not performed after adhesive application [41]. However, because it does not contain HEMA, it has very low water absorbency [41]. There are few studies on the adhesive properties of its successor, OptiBond eXTRa Universal [42,43], which probably shows high adhesion to enamel and dentin because it has the same basic composition as OptiBond XTR [42]. Our results showed that OptiBond eXTRa Universal exhibited high μTBS after 1 week of immersion in water, whether pre-treated with Silane Primer or OptiBond eXTRa Universal Primer. The results revealed that the RBC post-and-core build-up material also exhibited high adhesion without silane treatment.

OptiBond Universal, classified as a one-step one-bottle self-etch adhesive, is the successor to OptiBond All-in-One. Abdou et al. [44] showed that the μTBS of CAD/CAM RBC blocks bonded with NX3 after the application of OptiBond All-in-One to bovine dentin was significantly lower than that of Clearfil Universal Bond Quick and Panavia V5 (both from Kuraray Noritake Dental) or Scotchbond Universal Adhesive and Rely X Ultimate (both from 3M Oral Care). They speculated that this was due to the shorter and more hydrophilic spacer chains of GPDM, the adhesive monomer in OptiBond All-in-One, compared with MDP, the adhesive monomer in Clearfil Universal Bond Quick [44]. Our results showed that μTBS was higher than 50 MPa in the immediate group regardless of prior pre-treatment with Silane Primer. However, the values tended to be slightly lower than those of OptiBond eXTRa Universal and OptiBond Solo Plus. OptiBond Universal showed less adhesive failure than the other two products, while scratches that may have been formed by the SiC paper were clearly observed in the SEM images. OptiBond Universal contains water, acetone, and ethanol as solvents. If the volatilization of these solvents is insufficient, the adhesive itself cannot be cured even after light-curing [41]. Furthermore, sufficient air-drying to volatilize the solvents would cause a thin adhesive layer [44], and the greater standard deviation compared with OptiBond eXTRa Universal also suggests that adhesion is unstable.

OptiBond Solo Plus, classified as a two-step etch-and-rinse adhesive, has the longest history among the adhesives used in this study. In general, OptiBond FL, a three-step etch-and-rinse adhesive, and Clearfil SE Bond, a two-step self-etch adhesive, are known as gold standard adhesive systems [40]. OptiBond Solo Plus has been reported to exhibit high immediate adhesion to dentin, almost equal to these adhesives [45]. Furthermore, it has been used as an adjunctive adhesive in indirect restorations using Nexus 2 (Kerr), the previous generation of NX3 luting cement [46,47]. This adhesive shows high bond strength not only to dentin but also to enamel because of the presence of phosphoric acid [46]. In the present study, a very high initial bond strength was obtained without additional treatment with Silane Primer, suggesting that good adhesion to enamel, dentin, and RBC post-and-core build-up material of the abutment teeth can be obtained by treating them in the same manner.

In the present study, the aging of I-beam-shaped adhesive specimens by immersion in water for an additional 6 months significantly reduced the μTBS of all groups. It is generally believed that the decrease in bond strength is caused by hydrolysis resulting from inadequate chemical bonding at the adhesive interface. However, one of the main

causes of the decrease in bond strength in this study was assumed to be the degradation of the luting cement itself due to aging [48]. NX3 is a dual-cured composite cement, so mixing the two pastes results in a certain degree of curing without light-curing. However, its polymerization has been noted to be limited [49]. A 9 mm thick RBC CAD/CAM block was used in this study, and light-curing was performed for 20 s in each of four directions immediately after luting, which may have resulted in a higher polymerization rate of the luting cement at the edges. However, this light causes reflection and scattering inside the block, so very little light can penetrate [50,51]. The degradation of the luting cement may have been accelerated because all I-beam-shaped microtensile bond specimens were made after 1 week of immersion in water and were then aged again in water.

Another possible cause is the deterioration of adhesion between the RBC CAD/CAM blocks and the luting cement. Lührs et al. [29] investigated the pre-treatment of NX3 using LAVA Ultimate (3M Oral Care), an RBC CAD/CAM block. They reported that there was no significant difference between the results using Silane Primer and OptiBond XTR Adhesive before bonding to NX3 [29]. Capa et al. [36] also investigated the pre-treatment of NX3 using RBC CAD/CAM blocks. They reported that the use of both Silane Primer and OptiBond XTR after alumina airborne particle abrasion resulted in significantly higher bond strength than Silane Primer alone or OptiBond XTR alone [36]. Based on these previous studies, this study used Silane Primer followed by OptiBond eXTRa Universal after alumina airborne particle abrasion. However, Hagino et al. [52] reported that the bond strength of RBC CAD/CAM blocks with silane-containing surface treatment decreased after long-term immersion in water. In fact, the fine undulations observed with SEM in this study were similar to the delamination between RBC CAD/CAM blocks and luting cement reported by Lührs et al. [29].

We could not confirm the effectiveness of the additional silane treatment prior to the application of adhesive for the RBC for post-and-core build-up. Contamination of silane on the dentin surface has been reported to reduce bond strength [53]. Silane should not be applied to the built-up RBC prior to pre-treatment using adhesive, as it may compromise the clinical outcome. The design of this study assumed that luting would be performed relatively earlier after tooth preparation. Further investigation should be undertaken with optimal pre-treatment for aged RBC. Changes in adhesive behavior over a longer period of time must also be investigated.

5. Conclusions

Considering the limitations of the current in vitro study, we drew the following conclusions:

1. Additional silane treatment did not affect the microtensile bond strength of the resin-based composite for post-and-core build-up prior to the application of one-step self-etch adhesive, two-step self-etch adhesive, or two-step etch-and-rinse adhesive.
2. Additional silane treatment prior to the application of adhesive did not prevent the aging-induced loss in microtensile bond strength.

Since additional silane pre-treatment prior to the application of adhesive does not enhance the bond strength between resin-based composite post-and-core build-up material and luting cement, silane should not be applied to built-up resin-based composites, as it may compromise the clinical outcome.

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