Influence of resin-coating on bond strength of resin cements to dentin and CAD/CAM resin block in single-visit and multiple-visit treatment

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The purpose of this study was to evaluate the influence of resin-coating using one-bottle adhesives on the bond strength of resin cements in single-visit and multiple-visit treatments. Three one-bottle adhesives were used for resin-coating and/or pre-treatment adhesives prior to cementation, in conjunction with resin cements from the same manufacturers: Clearfil Universal Bond Quick with Panavia V5 (UBQ/Pv5), Scotchbond Universal Adhesive with RelyX Ultimate (SBU/RxU), and Optibond All-in-one with NX3 Nexus (OP/NX3). Bovine dentin surfaces were left uncoated or resin-coated. After 1-h water storage (single-visit) or 1-week water storage with a non-eugenol temporary cement (multiple-visit), a CAD/CAM resin block was cemented to uncoated or resin-coated dentin surfaces. Microtensile bond strengths (μTBSs) were measured and statistically analyzed (α=0.05). Application of resin-coating improved μTBSs. The multiple-visit group exhibited lower values of μTBS than the single-visit group. Selection of the materials affected μTBSs. Resin-coating and single-visit treatment are desirable for CAD/CAM resin composite restorations.

Keywords: Resin coating, Single visit, CAD/CAM, Microtensile bond strength, One-bottle adhesive

INTRODUCTION

Computer-aided design and computer-aided manufacturing (CAD/CAM) has become a major trend in restorative dentistry10). This technology has evolved as an alternative to conventional impression techniques, stone models/cast, and technician-produced restorations. In addition to the use of high-strength and glass ceramics, resin composite blocks have been widely used2). Owing to the industrial high-temperature/high-pressure polymerization and high filler loading, a significant improvement in their mechanical properties was achieved4). Resin composite blocks show equivalent fracture and fatigue resistance to glass ceramics4). Furthermore, they are easier to fabricate and repair and show less marginal chipping and wear to the opposing natural dentition than glass ceramics5,6).

Using digital scanning of the prepared tooth and chairside manufacturing of the restoration, the CAD/CAM technology enabled single-visit treatment5). Nevertheless, many clinical situations require multiple-visit treatment, which needs temporization with a provisional cement8). In the multiple-visit scenario, the temporary cement may remain on the dentin surface and affect the bond strength of the final cementation8).

In order to prevent tooth sensitivity and bacterial invasion of the highly permeable dentin exposed during preparation, the resin-coating technique was introduced in the 1990s10). In this technique, the combination of the dentin bonding agent and flowable resin composite was used to cover the prepared tooth surface before taking the impression. This technique also results in an improvement in bonding performance of resin cement and reduction in marginal leakage and gap formation11,12).

Resin-coating with a bonding agent and a flowable resin composite creates a thick layer (>100 μm) and deforms the prepared crown surface11,13). The one-bottle adhesive system is recommended for resin-coating for crown preparation14). Dual-application of all-in-one adhesive remarkably improves dentin bond strength and creates a layer less than 20 μm thick14). In routine dental practice, one-bottle adhesives, i.e., universal adhesives and all-in-one adhesives, have gained popularity due to their versatility and reliable bond strength15-17). Some one-bottle adhesives can be used for pre-treatment of direct and indirect restorations15). Thus, when a one-bottle adhesive is used for single-application of resin-coating and pre-treatment prior to cementation, one-bottle adhesive is applied twice17).

In this experiment, three one-bottle adhesives were used for resin-coating and/or pre-treatment adhesives prior to cementation in both single-visit and multiple-visit treatments. The aim of this study was to evaluate the influence of resin-coating using one-bottle adhesives and to compare single-visit and multiple-visit treatments on the bond strength of resin cement to dentin and CAD/CAM resin block. The three null hypotheses were tested: (i) application of resin-coating, (ii) single-visit...
or multiple-visit treatment, and (iii) selection of resin-coating material and resin cement did not affect the bond strength of resin cement to dentin and CAD/CAM resin block.

MATERIALS AND METHODS

The materials used in this study and their compositions and application procedures are presented in Table 1. Three one-bottle adhesives, two universal adhesives, and one all-in-one adhesive were used for resin-coating and/or adhesives prior to cementation, in conjunction with the dual-cure resin cements from the same manufacturers: Clearfil Universal Bond Quick with Panavia V5 (UBQ/Pv5, Kuraray Noritake Dental, Tokyo, Japan), Scotchbond Universal Adhesive with Relix Ultimate (SBU/RxU, 3M ESPE, St. Paul, MN, USA), and Optibond All-in-one with NX3 Nexus (OP/ NX3, Kerr, Orange, CA, USA). A eugenol-free temporary cement, Temp bond NE (Kerr) was used. A CAD/CAM resin block, Katana Avencia Block (Kuraray Noritake Dental) was used. A light-emitting diode (LED) light-curing unit (VALO, Ultradent, South Jordan, UT, USA) was used. A low-speed diamond saw (Isomet). The slabs were air-abraded using 50 μm Al2O3. The specimens were then ground with 600-grit SiC paper to expose the dentin surfaces. The specimens were then either left uncoated (non-coated) or resin-coated by one of three one-bottle adhesives: UBQ, SBU, and OP, according to the manufacturers’ instructions. The specimens were further divided into two subgroups: single-visit and multiple-visit subgroups. For the single-visit subgroup, the specimens were stored in distilled water at 37°C for 1 h. For the multiple-visit subgroup, each specimen was covered with eugenol-free temporary cement (Temp-Bond NE) and stored in water at 37°C. After one week, the temporary cement was removed from the surface with a spoon excavator and the surface was cleaned with alcohol-soaked cotton pellets for 10 s.

Regarding the CAD/CAM resin block preparation before cementation procedures, the Katana Avencia Block was sectioned into 1.5 mm thickness slabs using a low-speed diamond saw (Isomet). The slabs were air-abraded using 50 μm Al2O3 (Heraeus Kulzer Japan, Tokyo, Japan) under 0.1 MPa air pressure at 10 mm distance for 1 min and ultrasonically cleaned for 3 min in distilled water. Prior to cementation with Pv5 and RxU, the fitting surfaces of the CAD/CAM slabs were treated with UBQ and SBU (silane containing adhesives), respectively. Meanwhile, those cemented with NX3 were treated by 37.5% phosphoric acid (Gel etchant, Kerr) for 60 s, rinsed with water, air-dried, treated with a silane coupling agent (Silane Primer, Kerr), and air-dried again for 15 s.

Before cementation, one of three one-bottle adhesives was applied to the dentin surface and air-dried according to manufacturers’ instructions. For the resin-coated group, the same adhesive that was used for resin-coating was applied again to the specimens. The mixed cement paste of either Pv5, RxU, or NX3 was applied to the CAD/CAM slab. The CAD/CAM slab was then seated to dentin surface under finger pressure and light cured for 20 s.

After 24 h of water storage at 37°C, each specimen was sectioned vertically using a low-speed diamond saw in a mesiodistal and buccolingual direction to obtain beams with approximately 1×1 mm² cross-section. A digital caliper (Mitutoyo CD-15C, Mitutoyo, Kanagawa, Japan) was used to check beam dimensions before the µTBS test. Each specimen was tested for µTBS using a universal testing machine (EZ-SX, Shimadzu, Kyoto, Japan) at a cross-head speed of 1 mm/min after mounting in a testing jig (Danville engineering, San Ramen, CA, USA) with a cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan).

Failure mode analysis

Subsequent to µTBS test, fractured beams were examined by visual inspection first and then desiccated and gold-coated for a scanning electron microscope (SEM) (JSM-5310LV, JEOL, Tokyo, Japan) at 100× magnification. The fracture modes were classified into one of the five categories:

A: cohesive failure within dentin
B: adhesive failure along dentin interface
C: adhesive failure between resin cement and adhesive
D: adhesive failure between resin cement and CAD/CAM resin block
E: Pre-test failure.

Fourier transform infrared spectroscopy (ATR-FTIR) analysis

The infrared spectrum was analyzed with ATR-FTIR (Nicolet iS50 FT-IR spectrometer, Thermo Fisher Scientific, Waltham, MA, USA). The absorbance spectrum was acquired by thirty-two scans co-addition, a wavenumber range of 4,000 to 400 cm⁻¹ and a 4 cm⁻¹ resolution. The labial surfaces of the bovine incisors were ground with 600-grit SiC paper to expose a flat dentin surface. One of the three one-bottle adhesives was applied to dentin surfaces by lightly rubbing each dentin surface using a microbrush and the spectrum was acquired before light-curing. The spectra were recorded immediately after light-curing and after 1 h water storage at 37°C in a light-proof sealed container. Degree of conversion (DC) was calculated using the equation: DC=100×[(R<sub>r</sub>/R<sub>0</sub>)-(R<sub>r</sub>/R<sub>0</sub>)]<sup>-1</sup>, where R represents the peak of aliphatic C=C (around 1,638 cm⁻¹) and internal standard (reference peak, around 1,608 cm⁻¹) representing the aromatic double bond.18,19.
| Material | Code | Manufacturer | Composition | Application procedure | Batch No |
|----------|------|--------------|-------------|-----------------------|----------|
| Clearfil Universal Bond Quick | UBQ | Kuraray Noritake Dental, Tokyo, Japan | 10-MDP, Bis-GMA, HEMA, hydrophilic amide monomer, colloidal silica, ethanol, water, silane coupling agent, sodium fluoride, CQ (pH=2.3) | To dentin: Apply till wetting - Air-blow for 5 s. - Light cure for 10 s | 2L0024 |
| Scotchbond Universal Adhesive | SBU | 3M ESPE, St. Paul, MN, USA | 10-MDP, dimethacrylate resins, HEMA, methacrylate-modified polyalkenoic acid copolymer (Vitrebond™ Copolymer), filler, ethanol, water, initiators, silane (pH=2.7) | To dentin: - Apply and scrub for 20 s. - Air-blow for 5 s. - Light cure for 10 s To CAD/CAM: - Apply and scrub for 20 s. - Air-blow for 5 s. - Light cure for 10 s | 642539 |
| Optibond All-in-one | OP | Kerr, Orange, CA, USA | GPDM, mono- and di-functional methacrylate monomers, water, acetone, ethanol, CQ, fillers, sodium hexafluorosilicates and ytterbium fluoride (pH=2.5) | To dentin: - Apply and scrub for 20 s. - 2nd application and scrub for 20 s. - Air-blow for 5 s. - Light cure for 10 s To CAD/CAM: - Apply Silane Primer and scrub - Air-blow for 5 s - Apply OP and scrub for 20 s. - 2nd application and scrub for 20 s. - Air-blow for 5 s. | 6166766 |
| Kerr Silane primer | SP | Kerr | MPTMS, Bis-EMA, TEGDMA, ethanol Paste A: Bis-GMA, TEGDMA, hydrophobic aromatic-DM-A, hydrophilic aliphatic-DM-A, initiators, accelerators, silanated barium glass filler, silanated fluoroaliminosilicate glass filler, colloidal silica Paste B: Bis-GMA, hydrophobic aromatic-DM-A, hydrophilic aliphatic-DM-A, silanated barium glass filler, silanated aluminium oxide filler, accelerators, CQ, pigments Base: methacrylate monomers, radiopaque silanated fillers, initiator components, stabilizers and rheological additives | - Apply to the resin block and attach to tooth structure. - Light cure for 10 s. | 6193448 |
| Panavia V5 | Pv5 | Kuraray Noritake Dental | Bis-GMA, UDMA, EBPADMA, TEGDMA, inert mineral fillers, activators, stabilizers, radiopaque agent Base: zinc oxide, mineral oil, corn starch Catalyst: resin, ortho-ethoxybezoic acid, carnauba wax, octanoic acid | - Apply to the resin block and attach to tooth structure. - Light cure for 20 s. | 645829 |
| RelyX Ultimate | RxU | 3M ESPE | Bis-GMA, UDMA, EBPADMA, TEGDMA, inert mineral fillers, activators, stabilizers, radiopaque agent | - Apply to the resin block and attach to tooth structure. - Light cure for 20 s. | 6108661 |
| NX3 Nexus | NX3 | Kerr | Bis-GMA, UDMA, EBPADMA, TEGDMA, inert mineral fillers, activators, stabilizers, radiopaque agent | - Apply to the resin block and attach to tooth structure. - Light cure for 20 s. | 23335/6CU |
| Temp-Bond NE | — | Kerr | Bis-GMA, UDMA, EBPADMA, TEGDMA, inert mineral fillers, activators, stabilizers, radiopaque agent Base: zinc oxide, mineral oil, corn starch Catalyst: resin, ortho-ethoxybezoic acid, carnauba wax, octanoic acid | - Mix Base and Catalyst for 30 s. | 000320 |

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol-A-diglycidyl methacrylate; HEMA: 2-hydroxyethyl methacrylate; CQ: camphorquinone; GPDMD: glycerol phosphate dimethacrylate; MPTMS: γ-methacryloxypropyltrimethoxysilane; Bis-EMA: thoxylated bisphenol-A dimethacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: vurethane dimethacrylate; EBPADMA: ethoxylated bisphenol A-dimethacrylate
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Thicknesses of adhesive and resin-coating layer
Three specimens for each group were prepared in the same manner as for the μTBS test and stored in water at 37°C for 24 h. Bonded specimens were embedded in self-curing epoxy resin (Epoxide 2, Buehler) and sectioned into two halves using a low-speed diamond saw (Isomet). Finishing was done using a sequence of 600-, 800-, 1000-, 1200-, 1500-, and 2000-grit SiC papers under running water followed by diamond pastes down to 0.25 μm (DP-Paste P, Struers A/S, Ballerup, Denmark). In each step, specimens were cleaned ultrasonically. The specimens were kept in a desiccator for 24 h and then observed under a SEM (JSM-IT100, JEOL) at a 1,000× magnification. The thickness of adhesive and resin-coating layer was analyzed using ImageJ software (Version 1.52, National Institute of Health, Bethesda, Maryland, USA) at 15 different points for each sample and the average was used as sample mean.

Dentin surface examination after temporary cement removal
Three dentin specimens for each group were prepared in the same manner as for the μTBS test. Adhesive application was done according to the manufacturers’ instructions, followed by temporary cement application. After one week, the temporary cement was removed from the surface with a spoon excavator and the surface was cleaned with alcohol-soaked cotton pellets for 10 s. The specimen surfaces were examined using SEM (JSM-IT100) at a 270× magnification.

Statistical analyses
For μTBS data, the mean μTBSs calculated for each tooth were used for the analysis. The Kolmogorov-Smirnov and Shapiro-Wilk tests were used to check the normality of data distribution. The comparison among the tested groups in terms of “resin-coating”, “material”, and “visit” was performed using a three-way ANOVA. Post-hoc multiple pairwise comparisons with Bonferroni’s correction were then conducted (IBM SPSS Statistics for Windows, Version 23.0). The significance level of α=0.05 was used for all statistical analyses.

RESULTS

μTBS
The mean μTBS and standard deviations are presented in Table 2. The three-way ANOVA showed that three factors, resin-coating, materials, and visit, had a significant effect on mean μTBS (p<0.001), whereas their interaction was not significant (p=0.371). For the UBQ/Pv5 group, the non-coated group showed a significantly lower μTBS compared to the resin-coated group for both single-visit and multiple-visit treatments. For the SBU/RxU group, the resin-coating significantly increased the μTBS only for single-visit treatment. Single-visit/resin-coated group showed significantly higher μTBS compared to multiple-visit/non-coated group for OP/NX3. Regarding the comparison between materials, there were no significant differences between UBQ/Pv5 and SBU/RxU in all tested sub-groups except multi-visit/resin-coated group. UBQ/Pv5 showed significantly higher μTBS compared to OP/NX3 for all tested sub-groups.

Weibull characteristic strength (α), modulus (β), and the probability of failure (PI0) are presented in Table 3 and Fig. 1. The Weibull analysis showed that UBQ/Pv5 and SBU/RxU with resin-coating showed the highest characteristic strength among all the groups for the single-visit treatment. OP/NX3 with a non-coated/multiple-visit group showed the lowest characteristic strength and Weibull modulus among all the groups. Resin-coated groups showed significantly higher characteristic strengths than non-coated groups in the same materials and the same visiting groups except SBU/RxU with the multiple-visit group and OP/NX3 with the single-visit group. Single-visit groups revealed significantly higher characteristic strengths than

| Table 2 Results of the mean μTBS and standard deviations |
|---------------------------------|-----------------|-----------------|
|                                 | UBQ/Pv5         | SBU/RxU         | OP/NX3          |
| single-visit                    |                 |                 |                 |
| non-coated                      | 39.4±2.8a       | 36.4±2.4ab      | 27.2±4.1a       |
| resin-coated                    | 48.3±4.4ab      | 44.0±3.4ab      | 29.3±2.2a       |
| multiple-visit                  |                 |                 |                 |
| non-coated                      | 29.3±2.4ab      | 27.7±3.8a       | 22.1±3.7a       |
| resin-coated                    | 37.2±1.7ab      | 31.3±2.5ab      | 26.1±4.6ab      |

All values are given as the mean±SD. Different lowercase letter within each column indicates significant difference (p<0.05). Different uppercase letter within each row indicates significant difference (p<0.05).
Table 3  Weibull analysis and failure mode analysis for μTBS data

|                | α (95% CI)      | β (95% CI) | P10 (95% CI) | Failure mode (%) |
|----------------|-----------------|------------|--------------|------------------|
|                | single-visit    |            |              |                  |
| UBQ/Pv5        | non-coated      | 42.2 (39.7 to 44.8) | 5.9 (4.6 to 7.4) | 28.9 (25 to 32.2) | 23 34 32 11 0 |
|                | resin-coated    | 51.8 (48.8 to 54.8) | 6.1 (4.7 to 7.7) | 35.9 (31.2 to 39.9) | 11 26 52 11 0 |
| multiple-visit | non-coated      | 31.3 (29.7 to 32.8) | 7.1 (5.5 to 9.0) | 22.8 (20.1 to 25.0) | 6 23 68 3 0 |
|                | resin-coated    | 40.2 (37.6 to 42.8) | 5.5 (4.2 to 6.9) | 26.7 (22.8 to 30.1) | 20 20 46 14 0 |
| SBU/RxU        | non-coated      | 39.7 (36.6 to 42.9) | 4.6 (3.5 to 5.7) | 24.2 (20.0 to 27.9) | 11 29 43 17 0 |
|                | resin-coated    | 47.9 (44.1 to 51.8) | 4.7 (3.5 to 6.0) | 29.6 (24.3 to 34.3) | 3 29 52 9 9  |
| multiple-visit | non-coated      | 30.0 (27.8 to 32.4) | 5.0 (3.8 to 6.4) | 19.2 (15.9 to 22.0) | 11 43 31 3 11 |
|                | resin-coated    | 33.2 (31.6 to 34.7) | 7.8 (5.9 to 9.9) | 24.8 (22.1 to 27.1) | 9 26 51 11 3  |
| OP/NX3         | non-coated      | 29.9 (27.4 to 32.6) | 4.1 (3.2 to 5.2) | 17.4 (14.0 to 20.3) | 11 26 46 17 0 |
|                | resin-coated    | 31.0 (29.5 to 32.6) | 7.8 (5.8 to 9.9) | 23.2 (20.5 to 25.4) | 14 34 34 6 11 |
| multiple-visit | non-coated      | 23.6 (21.6 to 25.6) | 5.5 (3.9 to 7.5) | 15.7 (12.6 to 18.2) | 0 14 26 23 37 |
|                | resin-coated    | 29.0 (25.9 to 32.4) | 3.8 (2.8 to 5.0) | 16.1 (12.2 to 19.6) | 3 11 49 9 29 |

α: Characteristic strength, β: Weibull modulus, P10: Estimation and 95% confidence interval at 10% probability of failure. Different superscript letters within α column are statistically significant based on 95%CI. Different superscript letters within β column are statistically significant based on 95%CI.

A: cohesive failure within dentin; B: adhesive failure along dentin interface; C: adhesive failure between resin cement and adhesive; D: adhesive failure between resin cement and CAD/CAM resin block; E: Pre-test failure.

Fig. 1  Weibull Survival Plot with a horizontal reference line at 95% and 63.2% survival and vertical reference line at 30 MPa intersecting with survival curves. UBQ/Pv5 showed 100% survival with resin-coated and non-coated groups after single-visit treatment at 30 MPa. SBU/RxU showed 89% survival at 30 MPa for resin-coated groups in single-visit treatment and 66% survival in multiple-visit. OP/NX3 showed 53% survival in resin-coated/single-visit treatment at 30 MPa.

Regarding failure mode distribution, Types B and C were predominant failure modes in all groups except OP/NX3 with the multiple-visit group. OP/NX3 with the multiple-visit group showed 37% of Type E in the non-coated group and 29% of Type E in the resin-coated group.

**ATR-FTIR**

The results of DC are summarized in Table 4. A two-way ANOVA revealed that different adhesives and
Table 4  Degree of conversion (%)

|       | UBQ   | SBU   | OP    |
|-------|-------|-------|-------|
|       |       |       |       |
| immediate | 80.6±8.4<sup>a</sup> | 65.9±7.4<sup>bA</sup> | 77.4±3.2<sup>abA</sup> |
| 1 h    | 79.9±8.5<sup>a</sup> | 80.0±11.0<sup>bB</sup> | 88.3±4.0<sup>bB</sup> |

All values are given as the mean±SD. Different uppercase letter within each column indicates a significant difference (p<0.05). Different lowercase letter within each row indicates a significant difference (p<0.05).

Table 5  Thickness of adhesive layer (μm)

|       | UBQ   | SBU   | OP    |
|-------|-------|-------|-------|
|       |       |       |       |
| non-coated | 6.4±2.9<sup>a</sup> | 6.9±0.8<sup>a</sup> | 5.2±0.6<sup>a</sup> |
| resin-coated | 12.5±1.8<sup>b</sup> | 11.6±3.3<sup>b</sup> | 10.6±1.8<sup>b</sup> |

All values are given as the mean±SD. Different uppercase letter within each column indicates a significant difference (p<0.05).

Storage periods had a significant effect on DC% (p=0.021 and 0.008, respectively). Storage for 1 h resulted in a significant increase in DC% for SBU (p=0.007) and OP (p=0.032).

Thicknesses of adhesive and resin-coating layer
The thickness data are summarized in Table 5. Two-way ANOVA showed that only application of resin-coating had a significant effect on thicknesses of adhesive and resin-coating layer (p<0.001). On the other hand, the selection of all-in-one adhesives did not show a significant effect on thicknesses of adhesive and resin-coating layer (p=0.240). Non-coated groups showed 5.2–6.9 μm mean thickness of adhesives. Resin-coated groups showed around 10.6–12.5 μm mean thickness of adhesives. Representative cross-sectional images are shown in Fig. 2.

Dentin surface examination after temporary cement removal
The representative SEM images are shown in Fig. 3. Some debris was observed on both non-coated (Fig. 3(a)) and resin-coated (Fig. 3(b–d)) dentin after temporary cement removal.

DISCUSSION
The present study showed that resin-coating using the one-bottle adhesives improved the bond strength values for most of the tested groups, in agreement with...
the previous studies which used one-bottle adhesives or 2-step self-etch adhesives for resin-coating\(^1\)\(^2\)\(^3\). The resin-coating layer creates a low elastic modulus layer that works as a stress breaker or shock absorber\(^2\)\(^3\), thus resulting in higher bond strengths with resin-coated group.

When comparing the single-visit and multiple-visit treatments, there are two major differences: temporization and time from tooth preparation to final restoration. Single-visit treatment does not require temporization, as single-visit treatment enables the completion of a series of processes, including the preparation of a tooth, application of the resin-coating, impression-taking with an intraoral scanner, fabrication of restoration, and setting a final restoration within 1 h at the earliest\(^7\). Multiple-visit treatment, which is still frequently conducted requires at least 1 week for the completion of the restoration. Therefore, temporization is necessary for multiple-visit treatments in order to protect the prepared tooth and maintain the occlusion.

Several researches have evaluated the effect of temporization on dentin with/without resin-coating\(^9\)\(^2\)\(^4\)\(^2\)\(^5\). It was revealed that the removal of non-eugenol temporary cement with only the excavator was enough to avoid the reduction in the final bond strength\(^2\)\(^4\)\(^2\)\(^5\). However, cement remnants were observed in the non-coated and resin-coated groups (see Fig. 3). Frankenberger et al. reported that the temporary cement application showed a reduction in bond strength regardless of the removal method and that resin-coating prior to temporization increased the bond strength compared to without resin-coating\(^9\). Although temporary cements prevented dentin surface from direct water contact, there was microleakage between temporary cement and dentin\(^9\), which led to water uptake of the dentin and resin-coating layer.

The difference of elapsed time from tooth preparation to final restoration affects a few factors of the resin-coating layer i.e. degree of conversion and water sorption. As unreacted double bonds and free radical activity of resin-based materials decrease after polymerization, the possibility to obtain free radical polymerization and subsequent polymerization, with one-bottle adhesives used as pre-treatment materials for cementation, diminishes over time\(^2\)\(^7\). As the polymerizing network develops, the rate of radical propagation eventually becomes limited, even in the presence of unreacted monomer and free radicals\(^2\)\(^8\). In the present study, UBQ showed prominent degree of conversion immediately after light curing. Unlike SBU and OP, UBQ did not increase the degree of conversion between immediately after light curing and after 1 h water storage. As for water sorption, one-bottle adhesives uptake water over time, thus resulting in the degradation of mechanical properties of one-bottle adhesives. Hydrophilic amide monomer, which is contained in UBQ, contributes to show little water sorption and high mechanical property of UBQ\(^1\)\(^6\).

Among the tested materials, UBQ and SBU showed higher bond strengths than OP. This may be attributed to the presence of 10-MDP functional monomer in UBQ and SBU. It has been reported that 10-MDP forms a strong and reliable bonds with dentin\(^2\)\(^9\) by forming 10-MDP-Ca i.e. ‘nano-layering’ at the interface. On the other hand, OP contains GPDM functional monomer which exhibits a greater demineralization potential to dentin compared to MDP, which may result in efficient penetration of GPDM into deeply exposed collagen and a strong polymer network owing to its two methacrylate groups\(^2\)\(^0\). They may explain the insignificant difference in the bond strength between the non-coated and resin-coated groups for both single-visit and multiple-visit groups with OP/NX3. Nevertheless, GPDM-Ca salt is more susceptible to water degradation in comparison to the insoluble MDP-Ca salt\(^3\)\(^0\)\(^3\)\(^1\). This may explain the lower bond strength for OP/NX3 compared to UBQ/PV5 and SBU/RxU and the 30–40% incidence of pre-testing failure in OP/NX3 with the multiple-visit group. Previous research stated that the hydrophobicity and longer spacer chains of 10-MDP positively influenced the stability of the monomer-calcium ionic bonds\(^2\)\(^2\). On the contrary, the shorter and more hydrophilic spacer chains of GPDM might have caused overall reduction in the bonding of the GPDM containing adhesive after aging\(^2\)\(^0\). These facts are in agreement with the results of the Weibull analysis in the present study.

Regarding the thickness of one-bottle-adhesives, even resin-coated specimens showed approximately 11 to 13 μm thick layers on the dentin surface (see Fig. 2). This thickness will not affect the crown fitness, since resin-coating is applied prior to impression taking. However, the thickness of the adhesives can vary significantly according to the surface geometry in clinical situations. On smooth convex surfaces, the adhesive layer can be thin, while for concave regions such as marginal chamfer or shoulder, the adhesive layer might be thicker.

Fig. 3  SEM images (270× magnification) for representative dentin samples after temporary cement removal, arrow shows the remaining temporary cement after hand scaler removal.
(a) non-coated, (b) UBQ, (c) SBU and (d) OP.
than that obtained on the flat surface in the present study\textsuperscript{[33,34]}. In order to prevent the pool of adhesives around the margin, practitioners should be careful while air-blowing the one-bottle adhesives\textsuperscript{[35]}. In the current study, bovine teeth were used due to their availability and less variability in compositional structure. Despite the similarity between bovine and human teeth, human teeth preferable as it is more clinically relevant and this was a limitation of this study\textsuperscript{[36,37]}.

Based on the current results, the null hypotheses were rejected. We concluded that (i) application of resin-coating improved bond strength of resin cement to dentin and CAD/CAM resin block, (ii) single-visit treatment yielded higher bond strength of resin cement to dentin and CAD/CAM resin block than multiple-visit treatment, and (iii) selection of resin-coating material and resin cement affected the bond strength of resin cement to dentin and CAD/CAM resin block.

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