INTRODUCTION

Along with the rapid innovation in digital dentistry in recent years, a variety of chair-side CAD/CAM materials have been developed, such as polymer-infiltrated ceramic, resin nano ceramic, and composite blocks. The advantages of these newer materials over the commonly used feldspathic, leucite glass ceramic, and lithium disilicate glass ceramic CAD/CAM materials include less chipping during the milling procedure, resulting in better marginal adaptation, having elastic modulus closer to that of dentin, comparable wear properties to enamel, and suitability for intraoral repair. Additionally, in comparison with the direct and laboratory-processed composites, these materials have more favorable mechanical and physical properties, with less discoloration, higher abrasion resistance, and fewer voids, flaws, and cracks.

The new generation of polymer-based CAD/CAM materials are industrially polymerized at a higher temperature and pressure, and this high degree of polymerization necessitates adequate pre-treatment for bonding purposes. Creating a micro-retentive surface, by either sandblasting or tribochemical silica-coating followed by silanization, for chemical adhesion is beneficial for durable bonding of polymer-based CAD/CAM materials. Additionally, along with mechanical roughening, the hydrofluoric acid-silane interaction has demonstrated a significant beneficial effect on bonding to polymer-infiltrated ceramic.

The clinical success of adhesively bonded indirect restorations is primarily attributed to achieving a reliable bond between the restoration and the mineralized dental hard tissues, as well as to the micromechanical interlocking and chemical adhesion between the resin cement and restorative material. To establish a strong and durable bond, appropriate treatment of the respective surfaces is crucial. Moreover, selecting the appropriate resin cement can be a significant factor for achieving successful bonding of CAD/CAM restorations. The effectiveness of bonding different types of resin cements to tooth structure has been well-investigated. Similarly, different pre-treatment protocols for the new CAD/CAM materials are being intensively researched. However, the strength of the entire dentin-adhesive-resin cement-silane/adhesive-restorative material assembly contributes to the clinical success of restorations.

Therefore, the aim of this in vitro study was to evaluate the effects of two types of polymer-based CAD/CAM material (a resin nano ceramic and a composite), two types of resin cement (a self-etch resin cement with a two-step self-etch adhesive, and a self-etch resin cement with a universal adhesive), and different CAD/CAM material surface pre-treatments on the microtensile bond strength to dentin. The tested hypothesis was that the µTBS of the bonds formed between polymer-based CAD/CAM restorative materials and dentin is not influenced by the type of CAD/CAM material, resin cement, or the surface pre-treatment used.

MATERIALS AND METHODS

Two types of CAD/CAM material, a resin nano ceramic (Lava Ultimate [LVU], 3M ESPE, St. Paul, MN, USA) and a composite (CERASMArt [CS], GC, Tokyo, Japan),
two types of resin cement, a self-etch resin cement (NX3 Nexus Third Generation Universal Adhesive Resin Cement [NX3], Kerr, Orange, CA, USA) with a two-step self-etch adhesive (OptiBond XTR [OpXTR], Kerr) and a self-etch resin cement (RelyX Ultimate [RXU], 3M ESPE) with a one-step, self-etch universal adhesive (Single Bond Universal [SBU], 3M ESPE), and the respective silanes of the resin cements (Silane Primer, [SP], Kerr; RelyX Ceramic Primer [RXCP], 3M ESPE) were used in this study. The materials, their composition, and the application protocols are presented in Table 1.

Pre-treatment of the CAD/CAM specimens
The CAD/CAM blocks were cut into 4-mm-thick slabs with a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The surfaces to be bonded were then polished with 600-grit silicon carbide (SiC) paper, and sandblasted with 50-µm Al₂O₃ (Bego, Bremen, Germany) using a sandblasting unit (Basic Classic, Renfert, Hilzingen Germany) for 15 s at 0.2 MPa of pressure, at a distance of 10 mm from the target surface. Subsequently, the prepared CAD/CAM specimens were randomly divided into five groups according to which surface pre-treatment each group was to receive: SP, OpXTR adhesive, SP+OpXTR adhesive, SBU adhesive, or RXCP+SBU adhesive. All silane and adhesive applications on the CAD/CAM specimens were conducted according to the manufacturers' instructions; the adhesives applied to the CAD/CAM slabs were air-thinned, but not cured.

Preparation of dentin surfaces
Fifty extracted, non-carious human third molars were collected and used in this study according to a protocol approved by the Institutional Review Board of Yeditepe University (IRB No: 565). The teeth were stored in 0.5% chloramine-T solution at 4°C until their use. The teeth's occlusal enamel was removed using a slow-speed diamond saw (Isomet, Buehler) under water irrigation to create flat dentinal surfaces. A smear layer on the mid-coronal dentin (with 2–3 mm of dentin remaining) was created using a polishing machine (Buehler) at a rotation speed of 400 Rpm, with 600-grit SiC paper under running water.

Luting of the CAD/CAM specimens to dentin
During the luting procedure, adhesive tape (100 µm in thickness and 8 mm in diameter) was placed on the dentin surfaces of 50 specimens to standardize the resin cements' thickness. The LVU and CS specimens were bonded to the dentin surfaces with a self-etch approach using either NX3 with OpXTR (n=30) or RXU with SBU (n=20), according to the manufacturers' instructions. Following light curing of the OpXTR and SBU adhesives on the dentin surfaces, the respective resin cements, NX3 and RXU, were applied onto the LVU and CS specimens and luted to the dentin under a constant seating force of 1 kg16. The resin cements were immediately light-cured on the four proximal sides and top surfaces of all specimens, each for 20 s (for a total of 100 s per specimen), and constant pressure was applied for 5 min. Polymerization of the adhesive on the dentin surface and the resin cement was performed using an LED curing unit with a light intensity of 1,000 mW/cm² (Demi Plus, Kerr). The light intensity output was regularly checked with a radiometer throughout the entire luting procedure. Following curing, all bonded specimens were stored in distilled water at 37°C for 24 h, then submitted to thermal cycling for 5,000 cycles (5°C/55°C) with a dwell time in each water bath of 30 s and 5 s of transfer time using a thermocycling machine (Salubris-Technica, Istanbul, Turkey).

μTBS testing
After undergoing thermal cycling, the bonded specimens were sectioned into 1×1-mm sticks using a slow-speed diamond saw under water irrigation. From each tooth, five or six sticks were obtained from the center; to reduce bonding variation, the peripheral sticks were discarded. The remaining dentin thickness (2–3 mm) was determined with a digital micrometer. The sticks obtained from each group (n=22) were attached with cyanoacrylate glue (Zapit, DVA, Corona, CA, USA) to the Bencor Multi-T testing device, which was connected to a universal testing machine (Instron 3345, Norwood, MA, USA), then were subjected to microtensile testing at a crosshead speed of 1 mm/min. All bonded specimens were tested.

Failure mode analysis
After performing microtensile strength testing, the failure modes were determined with a stereo microscope (Leica MZ16 FA, Gantenbein, Switzerland) at 50× magnification to determine the fracture pattern. The debonded specimens were classified into one of five categories: cohesive failure within the CAD/CAM material, adhesive failure at the resin cement-CAD/CAM material interface, cohesive failure within the resin cement, adhesive failure at the dentin-resin cement interface, or mixed. Cohesive failures observed within the dentin were excluded from the study. Selected debonded specimens with characteristic fracture patterns from each group; were fixed for 24 h in 10% neutral buffered formalin, then placed on stubs, allowed to desiccate at room temperature, were gold sputter-coated, and observed with a scanning electron microscope (SEM; 6335-F, JEOL, Tokyo, Japan).

Statistical analysis
Statistical analyses were performed by three-way analysis of variance (ANOVA), using the types of CAD/CAM material, resin cement, and surface pre-treatment as independent variables with post-hoc Tukey’s tests (p<0.05; NCSS 2007 Statistical Software, NCSS, Kaysville, UT, USA).

RESULTS
The μTBS to dentin was significantly influenced by the types of CAD/CAM material (p<0.0001), surface
| Materials                  | Type                       | Manufacturer | Code     | Lot no.   | Application protocol                                                                                     | Composition                                                                                             |
|---------------------------|----------------------------|--------------|----------|-----------|---------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------|
| Lava Ultimate             | Resin nano ceramic         | 3M ESPE, St. Paul, MN, USA | LVU      | 5211595  | Sandblasting of specimen, further application steps                                                    | Nanoceramic resin ceramic (80%), Bis-GMA, UDMA, Bis-EMA, TEGDMA with silica nanomers (20 nm), zirconia nanomers (4–11 nm), nanocluster particles derived from the nanomers (0.6–10 µm), and silane coupling agents. |
| CERASMART                 | Composite CAD/CAM block    | GC, Tokyo, Japan | CS       | 1412092  | Sandblasting of specimen, further application steps                                                    | Composite resin (71%), Bis-MEPP, UDMA, DMA with nanoparticle-filled silica nanomers (20 nm), and barium glass (300 nm) filler. |
| Kerr Silane Primer        | Silane                     | Kerr, Orange, CA, USA | SP       | 5533000  | Apply Silane Primer to LVU and CS; after 60 s, dry with an air syringe (not light-cured)                | Ethyl alcohol and organosilane ester.                                                                     |
| OptiBond XTR Primer       | Self-etching primer        | Kerr         | OpXTR Pr | 5222982  | First, gently dry dentin. Apply primer for 20 s to dentin surface. Air thin for 5 s with medium air pressure. | Acetone, ethyl alcohol, and HEMA.                                                                           |
| OptiBond XTR Adhesive     | Adhesive                   | Kerr         | OpXTR Ad | 5234855  | Apply adhesive to dentin surface for 15 s. Air thin with gentle air first, then strong air for at least 5 s to avoid pulling. Light cure for 10 s. Apply adhesive to LVU and CS, and air thin (not light-cured). | Ethyl alcohol, alkyl dimethacrylate resins, barium alumino-borosilicate glass, fumed silica (silicon dioxide), and sodium hexafluoro-silicate. |
| NX3                       | Dual-cure resin cement     | Kerr         | NX3      | 5227499  | After completing bonding protocol on dentin surface, dispense cement with auto-mix syringe onto dentin surface. Light cure for 20 s. | Methacrylate ester monomers, inert mineral fillers, activators, stabilizers, and a radiopaque agent. |
| RelyX Ceramic Primer      | Silane                     | 3M ESPE      | RXCP     | N331797  | Apply silane to LVU and CS; after 60 s, dry with an air syringe.                                       | Ethyl alcohol, water, and methacryloxypropyltrimethoxysilane.                                                |
| Single Bond Universal     | Universal adhesive        | 3M ESPE      | SBU      | 665259   | Apply adhesive to dentin surface for 20 s. Gently air dry for 5 s. Light cure for 10 s. Apply adhesive to LVU and CS, and air thin (not light-cured). | Bis-GMA, HEMA, decamethylene dimethacrylate, ethanol, water, silane-treated silica, 2-propenoic acid, methacrylated phosphoric acid, copolymer of acrylic and itaconic acid, ethyl-4-dimethylaminobenzene, camphorquinone, (dimethylamino)ethyl methacrylate, and methyl ethyl ketone, silane. |
| RelyX Ultimate            | Dual-cure resin cement     | 3M ESPE      | RXU      | 641885   | After completing bonding protocol on the dentin surface, dispense cement with auto-mix syringe (clicker) onto dentin surface. Light cure for 20 s. | Base: Silane-treated glass powder, 2-propenoic acid, 2-methyl-1,1’-[1-(hydroxyethyl)-1,2-ethanediyl] ester, reaction products with 2-hydroxy-1,3-propanediyl dimethacrylate and phosphorus oxide, TEGDMA, silane-treated silica, oxide glass chemicals, sodium persulfate, tert-butyl peroxo-3,5,5-trimethylhexanoxoate, and copper (II) acetate monohydrate. Catalyst: Silane-treated glass powder, substituted dimethacrylate, 1,12-dodecanedimethacrylate, silane-treated silica, 1-benzyl-5-phenyl-barbic-acid, calcium salt, sodium p-toluene sulfonate, 2-propenoic acid, 2-methyl, and [3-methoxypropyl]iminodil-2,1-ethanediyl. | Bis-EMA: ethoxylated bisphenol-A-dimethacrylate; Bis-GMA: bisphenol-A-diglycidyl methacrylate; Bis-MEPP: 2,2-bis(4-methacryloxyethoxyphenyl) propane; DMA: dodecyl dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate. |
pre-treatment ($p<0.05$), and resin cement ($p<0.05$). Regardless of the surface pre-treatment method and type of resin cement used, the bond strength of CS to dentin was significantly higher than that of LVU ($p<0.0001$, Table 2). A significant interaction between surface pre-treatment and resin cement was found ($p=0.001$), while the interaction between CAD/CAM material and resin cement ($p=0.085$), CAD/CAM material and surface pre-treatment ($p=0.117$), CAD/CAM material/ surface pre-treatment/resin cement ($p=0.464$) were not significant.

For LVU and CS, the highest bond strengths were obtained when the CAD/CAM materials were pre-treated with SP+OpXTR adhesive (28.27±11.79 and 45.27±13.69 MPa, respectively), RXCP+SBU adhesive (28.85±7.58 and 48.6±11.83 MPa, respectively), and SBU adhesive (27.82±5.78 and 45.13±7.16 MPa, respectively), but were not significantly different from each other within each CAD/CAM material group ($p>0.05$). Conversely, the lowest µTBS's were observed when the CAD/CAM materials were pre-treated with SP (17.33±4.46 and 27.63±5.12 MPa, respectively) and OpXTR adhesive (18.56±5.6 and 29.72±5.49 MPa, respectively); the results for these pre-treatments were also not significantly different from each other within each CAD/CAM material

Table 2  Mean microtensile bond strength of adhesively bonded CAD/CAM materials to dentin (MPa, n=22)

| Surface pre-treatment of CAD/CAM materials | Resin cements                  | CAD/CAM materials |
|-------------------------------------------|--------------------------------|-------------------|
| Silane Primer (Kerr)                       | NX3/OptiBond XTR              | CERASMArt         | Lava Ultimate |
| OptiBond XTR adhesive (Kerr)               | NX3/OptiBond XTR              | 27.63±5.12 a, A   | 17.33±4.46 b, A |
| Silane Primer+OptiBond XTR adhesive (Kerr)| NX3/OptiBond XTR              | 29.72±5.49 a, A   | 18.56±5.6 b, A |
| Single Bond Universal (3M ESPE)            | RXU/Single Bond Universal     | 45.27±13.69 a, B  | 28.27±11.79 b, B |
| RelyX Ceramic Primer+Single Bond Universal (3M ESPE) | RXU/Single Bond Universal | 45.13±7.16 a, B   | 27.82±5.78 b, B |

NX3: NX3 Nexus Third Generation Universal Adhesive Resin Cement; RXU: RelyX Universal Cement.

Fig. 1  Scanning electron micrographs of untreated.
(a) and sandblasted Cerasmart specimens (b, c) (original magnifications: 5,000×, 1,000× and 5,000×, respectively).

Fig. 2  Scanning electron micrographs of untreated.
(a) and sandblasted Lava Ultimate specimens (b, c) (original magnifications: 5,000×, 1,000× and 5,000×, respectively).
Fig. 3  Failure mode analysis (CS: Cerasmart; LVU: Lava Ultimate; NX3: NX3 Nexus Third Generation Universal Adhesive Resin Cement; OpXTR: Optibond XTR; RLX: Rely X; RXCP: Rely X Ceramic Primer; SBU: Single Bond Universal).

Fig. 4 (a–c) Scanning electron micrographs of a fractured Cerasmart specimen that was pre-treated with Silane Primer and bonded with Optibond XTR and NX3 to dentin. Failure occurred adhesively at the CAD/CAM material-resin cement interface (original magnifications: 50×, 50× and 500×, respectively).

Fig. 5 (a–c) Scanning electron micrographs of a fractured Cerasmart specimen that was pre-treated with Optibond XTR adhesive and bonded with Optibond XTR and NX3 to dentin. Adhesive failure occurred at the Cerasmart-resin cement interface (original magnifications: 50×, 50× and 500×, respectively).
the CS specimens bonded with NX3 and the two-step self-etch adhesive OpXTR are shown in Figs. 4–6. The predominant failure mode that occurred on the CAD/CAM surfaces pre-treated solely with SP (Figs. 4a–c) and OpXTR adhesive (Figs. 5a–c) were adhesive failures between the resin cement and the CAD/CAM material. Conversely, the specimens treated with SP+OpXTR adhesive exhibited mostly mixed failure patterns occurring between the resin cement, adhesive, and dentin interface (Figs. 6a–c).

In contrast to those bonded with NX3, the CS specimens that were bonded with RXU+SBU, and pre-treated (on the CAD/CAM material’s surface) with either SBU-only (Figs. 7a–c), or RXCP+SBU (Figs. 8a–c) mostly exhibited mixed fracture patterns that occurred partially on top of the cement, within the adhesive, and at the dentin interface.

The failure modes of the LVU specimens bonded

![Fig. 6](a-c) Scanning electron micrographs of a fractured Cerasmart specimen that was pre-treated with Silane Primer+Optibond XTR adhesive and bonded with Optibond XTR and NX3 to dentin. Mixed failure occurred between the resin cement, adhesive and dentin interface (original magnifications: 50×, 50× and 500×, respectively).

![Fig. 7](a-c) Scanning electron micrographs of a fractured Cerasmart specimen that was pre-treated with Single Bond Universal and bonded with Single Bond Universal and Rely X Ultimate to dentin. Mixed failure occurred within the adhesive, resin cement and dentin interface (original magnifications: 50×, 50× and 500×, respectively).

![Fig. 8](a-c) Scanning electron micrographs of a fractured Cerasmart specimen that was pre-treated with Rely X Ceramic Primer+Single Bond Universal and bonded with Single Bond Universal and Rely X Ultimate to dentin. Mixed failure involving adhesive, resin cement and dentin was evident (original magnifications: 50×, 50× and 500×, respectively).
with NX3 and the two-step self-etch adhesive OpXTR are shown in Figs. 9–11. The failure modes observed with the LVU specimens that were bonded to dentin with NX3+OpXTR and pre-treated (on the CAD/CAM material’s surface) with either SP (Figs. 9a–c) or OpXTR adhesive (Figs. 10a–c) were mostly adhesive failures between the resin cement and the CAD/CAM material. In contrast, the specimens that were pre-treated with SP+OpXTR adhesive exhibited mixed failure patterns between the resin cement, adhesive, and dentin interface (Figs. 11a–c). The LVU specimens that were bonded with RXU+SBU, using either the SBU-only (Figs. 12a–c) or RXCP+SBU (Figs. 13a–c) pre-treatment on the CAD/CAM material’s surface, exhibited mostly mixed fracture patterns that occurred between the resin cement, adhesive, and dentin interface.

Fig. 9 (a–c) Scanning electron micrographs of a fractured Lava Ultimate specimen that was pre-treated with Silane Primer and bonded with Optibond XTR and NX3 to dentin, showing adhesive failure between the resin cement and CAD/CAM material (original magnifications: 50×, 50× and 500×, respectively).

Fig. 10 (a–c) Scanning electron micrographs of a fractured Lava Ultimate specimen that was pre-treated with Optibond XTR adhesive and bonded with Optibond XTR and NX3 to dentin. Adhesive failure occurring between the resin cement and CAD/CAM material was evident (original magnifications: 50×, 50× and 500×, respectively).

Fig. 11 (a–c) Scanning electron micrographs of a fractured Lava Ultimate specimen that was pre-treated with Silane Primer+Optibond XTR adhesive and bonded with Optibond XTR and NX3 to dentin, showing mixed failure pattern between resin cement, adhesive and dentin interface (original magnifications: 50×, 50× and 500×, respectively).
DISCUSSION

This in vitro study aimed to evaluate the effect of two different polymer-based CAD/CAM materials, their different surface pre-treatments, and use of different resin cements on the µTBS of adhesively bonded CAD/CAM onlays to dentin. The µTBS test was chosen as it allows a more uniform and homogeneous stress distribution compared to that of the conventional shear test, and it enables recognition of the weakest part of the adhesive complex based on the location of the failure.

Regarding the effects of different CAD/CAM materials on the µTBS, significant differences between LVU and CS were found (Table 2); thus, this study’s first null hypothesis was rejected. The difference in the chemical composition of various CAD/CAM materials may lead to variation in their mechanical properties and bonding performance. Although both CAD/CAM materials used in this study are polymer-based, CS is a ‘composite’ block that consists of evenly dispersed silica nanofillers and barium glass filler particles (71 wt%) and a low elastic modulus (12.2 GPa), whereas the nano ceramic LVU CAD/CAM material consists of silica nanomers (20 nm), zirconia nanomers (4–11 nm), and nanocluster particles derived from the nanomers (0.6–10 µm; 80 wt%) embedded in a highly cross-linked resin matrix. Both materials have similar flexural strength (LVU: 149–248 MPa) and elastic modulus (LVU: 13.8 GPa). However, LVU has a higher surface microhardness (97.9–102.3) than CS (62.2–64.1). Moreover, X-ray diffraction (XRD) analysis revealed that LVU contains crystalline ZrO₂, whereas CS contains non-crystalline resin and glass. Consequently, owing to the differences in filler-matrix composition and microhardness, CS may be more susceptible to mechanical roughening.

For adhesive bonding of polymer-based CAD/CAM materials, sandblasting with 50-µm Al₂O₃ at 0.2 MPa of pressure is recommended, which is much lower than the pressure applied to ceramics since these materials have lower microhardness. Sandblasting effectively provides a fresh, clean surface with increased surface roughness, surface area, and wettability, which make the surface more receptive for bonding. In this study, qualitative analysis using SEM revealed irregular surfaces for both CAD/CAM materials after sandblasting with 50-µm Al₂O₃ (at 0.2 MPa of pressure). However, rougher surfaces were observed for CS than LVU (Figs. 1 and 2), which is consistent with the lower microhardness of CS. These rougher surfaces may lead to possible increased micromechanical interlocking.
between the CS material and both types of resin cement, regardless of the surface pre-treatment method used, which may explain the higher $\mu$TBS's achieved with CS. In addition to this, in a previous study by Yoshihara et al., under the same sandblasting conditions cross-section SEM photomicrographs of the sandblasted LVU revealed several cracks within the large filler particles as well as the resin matrix while CS only showed a small crack near the surface, which suggests that this sandblasting condition may be too strong for LVU.

The surface pre-treatment methods applied to the CAD/CAM materials had a significant effect on the $\mu$TBS's in this study; hence, the second null hypothesis was also rejected. Application of silane followed by the adhesive, and the silane-containing universal adhesive with or without additional silane application were the most effective surface pre-treatment methods for both polymer-based CAD/CAM materials ($p<0.05$). Sandblasting exposes inorganic filler particles that become accessible for silanization. Silane application following sandblasting enables chemical adhesion; coupling between the inorganic filler particles of the CAD/CAM material and the inorganic and polymer components of the resin cement may enhance bond strength. The universal adhesive SBU contains a silane bi-functional monomer, which may be advantageous as it simplifies the bonding steps of the complicated adhesive luting procedure. Few studies have discussed the need for an additional silanization step when using a silane-containing universal adhesive for pre-treatment of polymer-based CAD/CAM materials. The results of the present study are in agreement with those of the studies conducted by Lührs et al.\(^{14,27}\); use of the silane-containing adhesive SBU with or without a separate silane primer for bonding to sandblasted LVU with RXU cement demonstrated similar $\mu$TBS’s to dentin after 1 week of storage. However, Yoshihara et al.\(^{29}\) evaluated the same adhesive with and without additional silane application for bonding to polished LVU and reported lower bond strengths for universal adhesive application only (after 15,000 thermocycles). The lack of micromechanical retention achieved through sandblasting and the longer thermocycling procedure used in that study may account for the difference in results when compared with our study. If the sandblasting step is omitted, the chemical bonding achieved with the silane incorporated into the universal adhesive may be insufficient, and additional silane application may be beneficial.

For both types of CAD/CAM materials, lower $\mu$TBS's were achieved with the use of the OpXTR adhesive or SP pre-treatments individually versus SP+OpXTR adhesive for the NX3 cement. These lower bond strengths were associated with adhesive failures at the resin cement-
CAM surface (Fig. 3), indicating that the hydrophobic OpXTR adhesive and SP pre-treatments were the weak links in the overall adhesive complex when they were used individually, and their consecutive applications (i.e., SP+OpXTR) contributed to durable resin-CAD/CAM material bonding. These results are consistent with the those reported by Lee et al., which demonstrated higher micro shear bond strength to lithium disilicate ceramic with NX3 when the ceramic surface was pre-treated with silane+porcelain bonding resin. However, mixed failures occurring between the CAD/CAM material-resin cement-adhesive and dentin interfaces were observed with high $\mu$TBS's when SP was applied followed by the OpXTR adhesive. A similar association was also observed for the RXU cement and the universal adhesive SBU, when either the RXCP followed by the SBU was applied to the CAD/CAM material’s surface or when SBU-alone was applied, which presented with 100% mixed failures (Fig. 3) and high bond strengths that are consistent with the data in the literature\(^{11,17}\).

The effects of the type of resin cement on the $\mu$TBS were less significant than the effect of the other two other factors investigated. The third null hypothesis, that there would be no significant differences in the $\mu$TBS's between the two resin cements’ (NX3 and RXU) bonds to dentin, was only accepted for the most effective CAD/CAM material surface pre-treatment methods: silane application followed by the adhesive (i.e., SP+OpXTR) or the universal adhesive application (i.e., SBU; $p>0.05$). For the other pre-treatment methods, use of RXU with the universal adhesive (i.e., RXU+SBU) resulted in higher $\mu$TBS to dentin ($p<0.05$) than NX3 with the two-step self-etch adhesive (i.e., NX3+OpXTR). According to the manufacturers’ instructions, both adhesives and cements should be light-cured separately and consecutively. A significantly higher degree of conversion and $\mu$TBS for LVU was documented with these two resin cements and the respective adhesives when they were separately light-cured during luting. By separately light-curing the adhesive, the adhesive interface and the hybrid layer on the dentin are stabilized. Based on the evidence that the bond strength to dentin was significantly reduced over storage in water for self-etch and etch-rinse adhesives, the adhesively bonded CAD/CAM specimens were thermocycled between water temperatures of 5 and 55°C for 5,000 cycles. Nonetheless, none of the specimens failed adhesively at the dentin-cement interface, indicating that durable dentin bonding with these two types of resin cements after thermocycling was achieved (Fig. 3).

In a previous study with a similar set-up, higher $\mu$TBS's for LVU CAD/CAM blocks were observed with NX3 and OpXTR than with SBU after 1 week of storage. The difference between these results and those of our study may be explained by the lack of aging. In this study, only a small number of failures occurred cohesively in the resin cement with NX3, and none occurred with RXU, indicating that both cements were sufficiently cured through the 4-mm-thick CAD/CAM slabs, and that the mechanical properties of the cured cements were sufficient to withstand the forces of the $\mu$TBS test (Fig. 3). Therefore, the pre-treatment methods seem more influential than the type of the resin cement for effective bonding of polymer-based CAD/CAM materials to dentin.
The design of this study has some limitations making it difficult to compare with long term clinical outcome. Longer aging periods with loading cycles would be more clinically relevant on evaluation of the tested parameters. Moreover, all the pre-treatment-methods were tested after only one micromechanical roughening approach; sandblasting. Different micromechanical and chemical bonding combinations such as tribochemical roughening and different types of silane containing universal adhesives would be beneficial to determine the interaction between the new polymer based CAD/CAM materials and the resin cements.

CONCLUSIONS

1) The type of CAD/CAM material used affected the µTBS’s of the resin cement-dentin bonds significantly. The composite CAD/CAM material appeared to be more receptive to bonding than the resin nano ceramic material.

2) Following sandblasting, chemical adhesion using silane along with a hydrophobic adhesive or a universal adhesive containing silane are both effective surface pre-treatment methods for both composite and resin nano ceramic CAD/CAM materials.

3) Following use of one of the most effective surface pre-treatment methods, the self-etch resin cement using a two-step self-etch adhesive, and the self-etch resin cement with a universal adhesive demonstrated similar bonding performances to dentin.

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