

## Original Article

### Effect of different adhesive strategies on microtensile bond strength of computer aided design/computer aided manufacturing blocks bonded to dentin

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#### ABSTRACT

**Background:** The aim of this study was to determine the microtensile bond strength ( $\mu$ TBS) of ceramic and composite computer aided design-computer aided manufacturing (CAD-CAM) blocks bonded to dentin using different adhesive strategies.

**Materials and Methods:** In this *in vitro* study, 30 crowns of sound freshly extracted human molars were sectioned horizontally 3 mm above the cemento-enamel junction to produce flat dentin surfaces. Ceramic and composite CAD/CAM blocks, size 14, were sectioned into slices of 3 mm thick. Before bonding, CAD/CAM block surfaces were treated according to the manufacturer's instructions. Groups were created based on the adhesive strategy used: Group 1 (G1) – conventional resin cement + total-etch adhesive system, Group 2 (GII) – conventional resin cement + self-etch adhesive system, and Group 3 (GIII) – self-adhesive resin cement with no adhesive. Bonded specimens were stored in 100% humidity for 24h at 37°C, and then sectioned with a slow-speed diamond saw to obtain 1 mm × 1 mm × 6 mm microsticks. Microtensile testing was then conducted using a microtensile tester.  $\mu$ TBS values were expressed in MPa and analyzed by one-way ANOVA with *post hoc* (Tukey) test at the 5% significance level.

**Results:** Mean values and standard deviations of  $\mu$ TBS (MPa) were 17.68 ( $\pm$ 2.71) for G1/ceramic; 17.62 ( $\pm$ 3.99) for G1/composite; 13.61 ( $\pm$ 6.92) for GII/composite; 12.22 ( $\pm$ 4.24) for GII/ceramic; 7.47 ( $\pm$ 2.29) for GIII/composite; and 6.48 ( $\pm$ 3.10) for GIII/ceramic; ANOVA indicated significant differences among the adhesive modality and block interaction ( $P < 0.05$ ), and no significant differences among blocks only, except between G1 and GII/ceramic. Bond strength of GIII was consistently lower ( $P < 0.05$ ) than G1 and GII groups, regardless the block used.

**Conclusion:** Cementation of CAD/CAM restorations, either composite or ceramic, can be significantly affected by different adhesive strategies used.

**Key Words:** Adhesion, ceramic, composite resin, computer aided design/computer aided manufacturing

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## INTRODUCTION

Tooth-colored ceramic restorations are becoming increasingly popular worldwide. Computer aided design/computer aided manufacturing (CAD/CAM) materials and chair-side machines that are capable of fabricating such restorations are of a particular interest so that a single dental appointment often can suffice to deliver the restoration.<sup>[1-3]</sup>

CAD/CAM ceramic restorations are simply bonded to teeth previously prepared to receive crowns, inlays, or onlays.<sup>[4]</sup> Cavity mechanical retention is usually not required once the primary source of retention is based on the adhesive interaction with the tooth substrate.

Currently, CAD/CAM blocks are available in a variety of different materials, shades, and sizes. The most common type of CAD/CAM block (feldspathic ceramic blocks) has been the subject of investigation in several studies, where strength,<sup>[5,6]</sup> color stainability,<sup>[7]</sup> margin fit,<sup>[8-10]</sup> and other parameters were evaluated. The newly emergent leucite glass ceramic and lithium disilicate ceramic blocks are gaining popularity due to better mechanical and aesthetic properties when compared to the feldspathic ceramic.<sup>[11-13]</sup> In addition to ceramic materials, CAD/CAM restorations also can be milled from composite blocks<sup>[4]</sup> and from a hybrid of composite resin and ceramic.<sup>[14]</sup> The ceramic blocks are manufactured by Vita and are a mixture of 30% by weight feldspathic crystalline particles embedded in a glassy matrix. It has a flexural strength of 112 MPa and a Young's modulus of 63 GPa.<sup>[15]</sup> Long-term clinical studies of inlays and onlays fabricated with this material have shown survival rates of 95% over 10-year of clinical life.<sup>[16]</sup> The second type of CAD/CAM block (composite blocks) are manufactured by 3M ESPE and they are made of 3M Z100 restorative material with 85% by weight ultrafine zirconia-silica ceramic particles that reinforce a highly cross-linked polymeric matrix based on Bisphenol A diglycidyl ether dimethacrylate and (tri[ethyleneglycol] dimethacrylate). It has a flexural strength of 109 MPa and a Young's modulus of 8 GPa.<sup>[4]</sup>

After completion of the tooth milling process, two separate adhesive treatments are utilized to cement the restorations. First, the inner treated surface of the restoration receives the adhesive system and the luting agent. The inner surface restoration treatments vary depending on the restoration material. For example,

ceramic restoration requires 30 s etching using 10% hydrofluoric acid on the ceramic inner surface to create a micro-retentive surface. A silane coupling agent is then applied to the restoration in order to create a chemical bonding with the resin-based luting cement. For the composite restoration, a sandblasting technique with aluminum oxide (50  $\mu$ m) is generally the only necessary surface treatment for this type of restorative material. On tooth surface, total-etch or self-etch adhesive techniques are employed to create an interaction with the resin luting agent. Either total-etch or self-etch adhesive techniques aim to create hybrid layer on the dentin surface. The so-called self-adhesive resin cement does not require a separate phosphoric acid application; however, previous studies have reported low bond strength on enamel and particularly on dentin.<sup>[17]</sup> Therefore, an understanding of the effects of different strategies of adhesion when cementing different CAD/CAM restoration is of paramount clinical importance and not fully explored in the literature.

This study was designed to determine the microtensile bond strength of ceramic and composite CAD-CAM blocks bonded to dentin using three different adhesive strategies: (i) Conventional resin cement with total-etch adhesive technique; (ii) conventional resin cement with self-etch adhesive technique; and finally (iii) self-adhesive resin cement with no adhesives. First, we determined whether the bond strength of CAD/CAM ceramic and composite blocks are influenced by the different adhesive strategies, and second, we examined if the type of CAD/CAM block differs in terms of bonding adhesion, regardless of the adhesive strategy used.

## MATERIALS AND METHODS

In this *in vitro* study, 30 freshly extracted human molars were sterilized via gamma radiation. Crowns of selected sound third molars were sectioned horizontally 3 mm above the cemento-enamel junction to expose sound dentin on a flat surface. The sectioned teeth were examined microscopically under  $\times 10$  magnification to ensure the absence of defects or caries, and then stored for 24 h in a temperature-controlled (37°C) incubator (Boeckel Analog Model 139400, PA, USA) with 100% humidity to avoid dehydration.

The CAD/CAM ceramic (Vita Mark II, Vita, Postfach, Germany) and composite blocks (Paradigm,

3M ESPE, St. Paul, MN, USA) were sectioned into 3 mm thick slices using a slow-speed saw equipped with a water-cooled diamond blade (IsoMet 1000–Buehler, Lake Bluff, IL, USA). For the ceramic blocks, one side was treated with 5% hydrofluoric acid (Cerec Ceramic Etch, Vita, CA, USA) for 30 s, then rinsed and air-dried. Two layers of silane coupling agent (Monobond Plus – Ivoclar Vivadent, Amherst, NY, USA) were applied to the etched surface for 30 s using a microbrush (Microbrush International, Grafton, WI, USA) and air-dried. For the composite block, one side was sandblasted with a 50 µm aluminum oxide jet (A.E. Aubin 1400 RC, New Milford, CT, USA) for 5 s and cleaned in an ultrasonic unit for 5 min (Branson Ultrasonic Cleaner, Danbury, CT, USA).

For this study, three different adhesive strategies were selected: (i) Conventional resin cement with total-etch adhesive system (Calibra + conventional bottle Primer and Bond NT associated with primer

and Bond activator - Dentsply) and labelled Group 1 (GI) ( $n = 30$ ); (ii) conventional resin cement with a self-etch adhesive system (Panavia F2.0 + Clearfil SE Bond - Kuraray) and called Group 2 (GII) ( $n = 30$ ); (iii) self-adhesive resin cement without adhesive system (SmartCem2 - Dentsply) called Group 3 (GIII) ( $n = 30$ ).

For GI, teeth were etched with 34% phosphoric acid (Caulk Tooth Conditioner Gel 34%, Dentsply Caulk, Milford, DE, USA) for 30 s (enamel) and 15 s (dentin) and then rinsed for 30 s with distilled water. Both enamel and dentin were air-dried, avoiding complete dehydration of the surfaces. Adhesive system and resin cement were mixed and applied following the manufacturer’s instructions [Table 1].

For GII, enamel and dentin etching techniques with phosphoric acid were not necessary. Self-etch adhesive and resin cement were applied according to the manufacturer’s instructions.

**Table 1: Resin cements tested in this study**

Resin cement	Manufacturer	Composition	Application protocol
Primer and Bond NT with self-cure Activator	Dentsply Caulk, Milford, DE, USA	Adhesive: Di- and trimethacrylate resins PENTA; nanofillers-amorphous silicon dioxide photoinitiators, stabilizers cetylaminehydrofluoride, acetone Self-cure activator: UDMA; HEMA; catalyst; photoinitiators; stabilizers; acetone; water	Apply 34% phosphoric acid on enamel and dentin, blot drying with air, then adhesive mixed with activator is applied on the tooth surface, air jet for 5 s and light-cured for 10 s
Calibra	Dentsply Caulk, Milford, DE, USA	Base paste: Bis-GMA; ethoxylatedbisphenol a dimethacrylate; 2,2'-ethylendioxydiethyldimethacrylate; dimethacrylate resins; CQ; photoinitiator; stabilizers; glass fillers; fumed silica; titanium dioxide; pigments; peroxide catalyst Catalyst paste: Bis-GMA; ethoxylatedbisphenol a dimethacrylate; 2,2'-ethylendioxydiethyldimethacrylate dibenzoyl peroxide; dibenzoyl peroxide	Dispense same amount of base and catalyst pastes onto a mixing pad and mix the cement for 20-30 s before applying on the tooth
Clearfil SE Bond	Kuraray Medical, Okayama, Japan	Adhesive: 10-methacryloyloxydecyl dihydrogen phosphate; hydrophobic aliphatic methacrylate; colloidal silica; dl-CQ; initiators; accelerators, others Primer: HEMA; 10-methacryloyloxydecyl dihydrogen phosphate; hydrophobic aliphatic dimethacrylate; dl-CQ; water; accelerators; dyes, others	Apply primer and leave for 20 s; dry with mild air flow; apply adhesive, air-dry and light cure for 10 s
Panavia F 2.0	Kuraray Medical, Okayama, Japan	Paste A: 10-methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic methacrylate, hydrophilic aliphatic dimethacrylate, silanated silica filler; silanated colloidal silica; dl-CQ; catalysts, initiators Paste B: Sodium fluoride; hydrophobic aromatic dimethacrylate; hydrophobic aliphatic methacrylate; hydrophilic aliphatic dimethacrylate; silanated barium glass filler; catalysts; accelerators; pigments others	Dispense same amount of base and catalyst pastes onto a mixing pad and mix the cement for 20-30 s before applying on the tooth
SmartCem 2	Dentsply Caulk, Milford, DE, USA	Barium boron fluoro alumino silicate glass; UDMA resin; urethane modified bis-GMA dimethacrylate resin; polymerizable dimethacrylate resins; hydrophobic amorphous silica	Install a mixing tip and gently depress the syringe plunger to start material flow; gently apply a thin uniform layer of cement to the entire internal surface of the restoration; seat the restoration, light cure for 20-40 s

PENTA: Dipentaerythritol pentaacrylate monophosphate; UDMA: Urethane dimethacrylate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: Bisphenol A-glycidyl methacrylate; CQ: Camphorquinone

For GIII, neither enamel nor dentin were etched or received adhesive application. The resin cement was mixed and applied following the manufacturer's instructions.

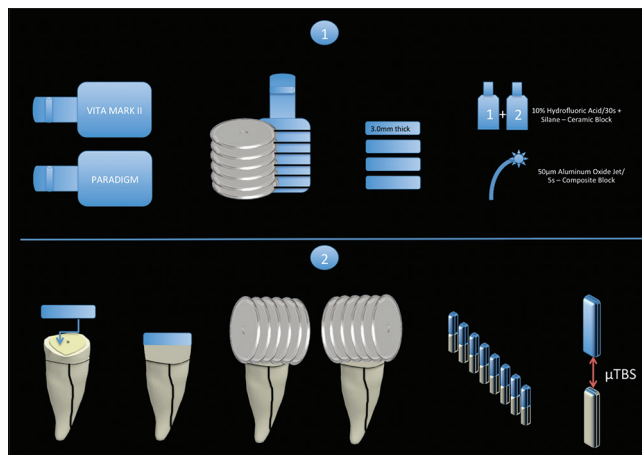
For all groups, ceramic and composite CAD/CAM block slices were bonded to the teeth under a constant seating force of approximately 50 N for 5 min using a special device designed to simulate finger pressure and equipped with a 5 kg weight whereas a high power L.E.D. light curing unit (SmartLite Max LED Curing Light, Dentsply Caulk, Milford, DE, USA) was used to cure the resin cements. Each surface of the teeth (buccal, lingual, mesial, and distal) was exposed to the light for 20 s. Then, teeth were stored in distilled water for 24 h at 37°C.

The teeth were then sectioned into rectangular microsticks (6 mm × 1 mm × 1 mm) using a slow-speed saw with a water-cooled 40 micron diamond blade (MTI, Richmond, CA, USA). Only microsticks from the most central portion of the dentin were used. The microsticks were bonded onto a specially designed metal apparatus using all-purpose Super Glue (Loctite, Westlake, Ohio, USA) and placed onto a tensile tester machine (BiscoMicrotensile Tester T-61010K, Schaumburg, Illinois, USA), where the bond strength was tested with a cross speed of 0.5 mm/min. Thirty unbroken microsticks were tested per cement group ( $n = 90$ ). Using a stereomicroscope at × 35 magnification (Nikon SMZ445, Melville, NY, USA) microsticks were also submitted for a failure analysis [Figure 1].

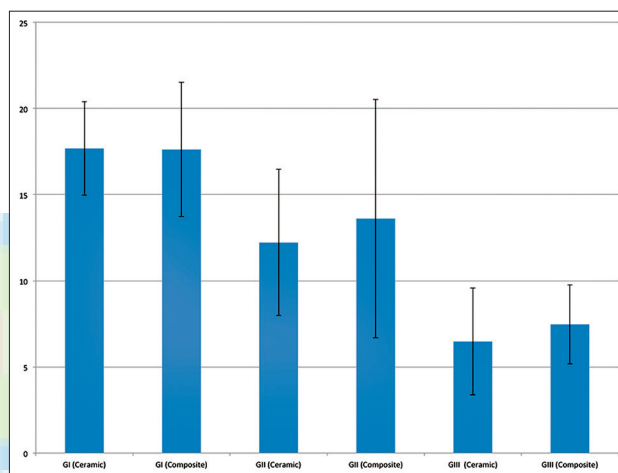
Microtensile data were expressed in Mega Pascal (MPa), by dividing the load at failure (Newton) to the bonding surface area (mm<sup>2</sup>) and analysed by a Statistical Software (SPSS version 22.0, IBM Software, Armonk, NY, USA) using two-way ANOVA with *post hoc* (Tukey) test at the 95% confidence.

## RESULTS

The  $\mu$ TBS mean values, standard deviations, and results of comparisons among different interactions are presented in Table 2 and statistical analysis (two-way ANOVA) in Table 3. ANOVA revealed significant differences among the adhesive strategy groups and adhesive strategy + blocks interaction ( $P < 0.05$ ); graphically presented in Figure 2 and then the Tukey *post hoc* test was applied to identify whether these differences occur. No significant differences between types of blocks only were found, except



**Figure 1:** Steps for fabrication of the rectangular microsticks (ceramic and composite).



**Figure 2:** Bond strength results for different adhesive strategies used.

**Table 2: Mean bond strength values (±SD) of investigated adhesive strategies and CAD/CAM blocks**

Adhesive strategy	Ceramic block	Composite block
Group 1	17.68 (±2.71)	17.62 (±3.90)
Group 2	12.22 (±4.24)	13.61 (±6.92)
Group 3	6.48 (±3.10)	7.47 (±2.29)

SD: Standard deviation; CAD/CAM: Computer-aided design/computer-aided manufacturing

when the comparisons are among the strategy GII/ceramic block and strategy GI/ceramic and composite blocks. The GII showed better interaction when used with CAD/CAM composite block. Bond strength of GII was consistently lower than GI group regardless of the block used; however, no statistical significance ( $P > 0.05$ ) was found when strategy GII/composite blocks only was compared with GI/ceramic or composite blocks; consequently, the strategy



**Table 3: Two-way ANOVA values**

Tests of between-subjects effects						
Dependent variable: Micro Tensile Bond Strength						
Source	Type III sum of squares	df	Mean square	F	Significant	Partial eta squared
Corrected model	11.482 <sup>a</sup>	5	2.296	9.027	0.000	0.350
Intercept	96.348	1	96.348	378.717	0.000	0.818
Cements	11.357	2	5.679	22.321	0.000	0.347
Blocks	0.002	1	0.002	0.008	0.930	0.000
Cements×blocks	0.123	2	0.061	0.241	0.786	0.006
Error	21.370	84	0.254			
Total	129.200	90				
Corrected total	32.852	89				

<sup>a</sup> $R^2=0.350$  (adjusted  $R^2=0.311$ )

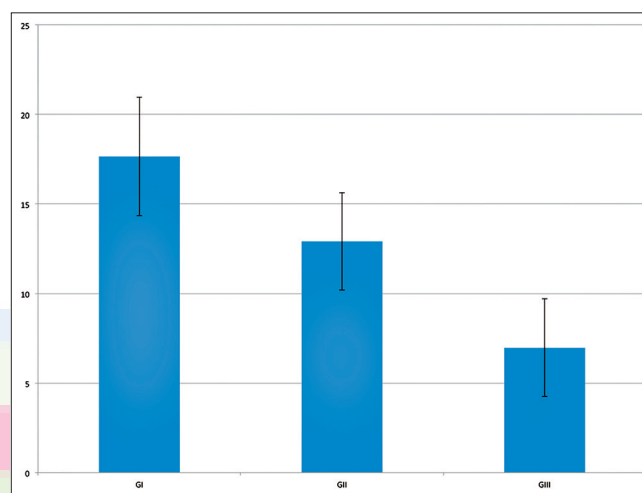
GII/ceramic blocks showed statistical significant differences when compared with strategy GI. Furthermore, the strategy GIII, which was consistently lower than GII, showed the statistical significant difference ( $P < 0.05$ ), regardless of the block used; graphically represented in Figure 3. For all groups, specimens failed predominantly at the interface resin cement-dentin.

## DISCUSSION

This research was designed to determine if the bond strength of CAD/CAM ceramic and composite blocks to dentin is influenced by different adhesive strategies. Our results show that these strategies do in fact influence bond strength. Based on this study, the conventional adhesive method using resin cement associated with a total-etch adhesive system is still the best option when cementing CAD/CAM restorations.

The second question was whether the different types of CAD/CAM block differ in terms of bonding adhesion, irrespective of the adhesive strategy used. The type of block turned out to be irrelevant.

One of the main advantages of CAD/CAM technology is the fact that restorations can be delivered in a single dental appointment avoiding the necessity of temporary restorations, final impressions, and lab phase. Clinicians are always looking for a simpler and faster bonding technique when dealing with cementation of indirect restorations. In our study, the total-etch adhesive (GI) showed the best values in terms of bonding adhesion when used with resin cement. However, this technique is time consuming requiring the application of different products onto the tooth surface before seating the restoration.



**Figure 3:** Comparison of evaluated adhesive strategies only.

Newer self-adhesive resin cement have the potential to simplify into a single-step the bonding process to cement indirect restorations. These new cement introduced successful integration of components from different material classes such as glass ionomer and composite resin cement technologies. Self-adhesive materials are based on a great quantity of acidic methacrylate monomers with phosphoric acid groups and reactive carbon double bonds.<sup>[17]</sup> Once in contact with the tooth structure, these phosphoric acid groups of the methacrylate monomers bond to  $Ca^{2+}$  ions in the tooth structure. At the same time, single monomer molecules are chemically cross-linked to form a polymer network, completing the polymerization reaction.<sup>[18]</sup> However, the use of self-adhesive resin cement to bond indirect restoration is controversial. Some authors believe that the methacrylate phosphoric esters cannot adequately penetrate the partly dissolved smear layer retained on the dentin, resulting in interfacial gaps and, consequently, lower bond strength.<sup>[19]</sup>

Although GII seems to be more successful than the GIII strategy in terms of bonding adhesion, previous studies have shown that the use of self-etching primers may behave as permeable membranes after polymerization, because of their higher concentration of hydrophilic and ionic resin monomers.<sup>[20]</sup> These studies also suggested the desirability of a subsequent application of a more hydrophobic resin coating layer on top of the self-etch adhesive in order to decrease the permeability and water diffusion from an adhesive-composite interface. Furthermore, other authors reported that the 10-MDP phosphate-based functional monomer present in the Clearfil SE bond system showed a good chemical interaction with the hydroxyapatite left around the collagen within the hybrid layer and this interaction appears to be important for long-term adhesive stability.<sup>[21]</sup> Therefore, the bonding adhesion between MDP and hydroxyapatite is stable due to the low solubility of the MDP-calcium salt in water.<sup>[22]</sup> In contrast, some authors have reported that self-etch adhesive systems have low interaction with enamel hydroxyapatite and an additional pretreatment of the enamel with 37% phosphoric acid for 30 s before adhesive application should be performed.<sup>[23,24]</sup> Although our study was limited to only dentin surface, further studies on enamel should be performed using the same methodology.

Our study also showed that composite and ceramic blocks have similar bonding adhesion with resin cement, regardless the adhesive strategy used. Both ceramic and composite surface treatment indeed improved the surface roughness and interaction with the resin cement.

## CONCLUSION

In light of the fact that differences in bonding strategies can interfere with the survivability of CAD/CAM restoration, our results should be tested in a clinical setting in future studies. Finally, cementation of CAD/CAM restorations, either composite or ceramic, can be significantly affected by different adhesive strategies.

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### Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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