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<td>Author(s)</td>
<td>Kameyama, A; Bonroy, K; Elsen, C; Lührs, AK; Suyama, Y; Peumans, M; Van, Meerbeek; De Munck, J</td>
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Original article

Luting of CAD/CAM ceramic inlays: direct composite versus dual-cure luting cement

Atsushi Kameyama a,b, Kim Bonroy c, Caroline Elsen c, Anne-Katrin Lührs a,d, Yuji Suyama a,e, Marleen Peumans a, Bart Van Meerbeek a and Jan De Munck c

a KU Leuven BIOMAT, Department of Oral Health Sciences, Biomedical Sciences Group, KU Leuven (University of Leuven), Kapucijnenvoer 7, B-3000 Leuven, Belgium

b Division of General Dentistry, Department of Clinical Oral Health Science, Tokyo Dental College, 2-9-18 Misaki-cho, Chiyoda-ku, Tokyo 101-0061, Japan

c Section of Operative Dentistry, School of Dentistry, Oral Pathology, and Maxillo-facial Surgery, KU Leuven (University of Leuven), Kapucijnenvoer 7, B-3000 Leuven, Belgium

d Department of Conservative Dentistry, Periodontology and Preventive Dentistry, OE 7740, Hannover Medical School, Carl-Neuberg-Straße 1, 30625 Hannover, Germany

e Toranomon Hospital, Department of Dentistry, 2-2-2 Toranomon, Minato-ku, Tokyo 105-8470, Japan

Running short head: Luting of CAD/CAM ceramic inlays
**Corresponding author:** Atsushi Kameyama, Division of General Dentistry, Department of Clinical Oral Health Science, Tokyo Dental College, 2-9-18 Misaki-cho, Chiyoda-ku, Tokyo 101-0061, Japan. Tel: +81-3-6380-9127; E-mail: kameyama@tdc.ac.jp

**Abstract.** The aim of this study was to investigate bonding effectiveness in direct restorations. A two-step self-etch adhesive and a light-cure resin composite was compared with luting with a conventional dual-cure resin cement and a two-step etch and rinse adhesive. Class-I box-type cavities were prepared. Identical ceramic inlays were designed and fabricated with a computer-aided design/computer-aided manufacturing (CAD/CAM) device. The inlays were seated with Clearfil SE Bond/Clearfil AP-X (Kuraray Medical) or ExciTE F DSC/Variolink II (Ivoclar Vivadent), each by two operators (five teeth per group). The inlays were stored in water for one week at 37°C, whereafter micro-tensile bond strength testing was conducted. The micro-tensile bond strength of the direct composite was significantly higher than that from conventional luting, and was independent of the operator ($P < 0.0001$). Pre-testing failures were only observed with the conventional method. High-power light-curing of a direct composite may be a viable alternative to luting lithium disilicate glass–ceramic CAD/CAM restorations.
Keywords: Micro-tensile bond strength, Ceramic inlay, Computer-aided design/computer-aided manufacturing (CAD/CAM), Luting cement, Self-etch
1. Introduction

The increasing demand for esthetic, tooth-colored restorations with reliable clinical performance can be fulfilled partly by indirect computer-aided design/computer-aided manufacturing (CAD/CAM)-restorations. This technology allows a ceramic restoration to be prepared, designed and fabricated in a single appointment, which eliminates the need for impression-taking and provisional restorations and prevents contamination of adhesive surfaces by temporary cements [1].

Stable adhesion to CAD/CAM glass ceramics can be obtained using resin luting cements to produce a combination of micro-mechanical interlocking and chemical adhesion by silanization [2-4]. Adhesion to the tooth substrate is, however, more challenging and weaker than that with the adhesive systems used in direct composite restorations [5,6]. Moreover, a general distinction can be made between easy-to-use but low-performing cements such as automix self-adhesive cements, and high-performing cements that require multiple application steps [5,7]. Direct light-cure restorative composites are perhaps considered more user-friendly because of the prolonged processing time required. The higher filler content and lower initiator concentration compared with dual-cure resin cements may be beneficial in terms of
mechanical strength and the wear properties of the exposed margins [8].

The objective of this study was to assess bonding effectiveness in direct restorations by comparing a gold-standard two-step self-etch adhesive and a light-cure resin composite with luting with a conventional dual-cure resin cement and a two-step etch and rinse adhesive. The null hypothesis tested in this study was that there was no significant difference in bonding effectiveness between the two procedures in the cementation of CAD/CAM ceramic blocks to Class-I cavity-bottom dentin.

2. Materials and Methods

2.1. Preparation of cavity and ceramic inlay

Twenty non-carious human third molars were harvested after extraction with informed consent. The study protocol was approved by the Commission for Medical Ethics of the University of Leuven. The specimens were stored in 0.5% chloramine T solution at 4°C and used within 1 month of extraction. Each specimen was mounted on a gypsum block to facilitate handling. A standard box-type Class-I cavity (approximately $4 \times 4$ mm) was prepared to a depth of 2.5 mm using a 1:5 high-speed motor hand-piece (INTRAmatic 25CH, KaVo Dental GmbH, Biberach, Germany) with a tapered cylindrical
medium-grit (100 µm) diamond bur (848 314 023, Komet, Lemgo, Germany) mounted in a modified computer-controlled MicroSpecimen Former (University of Iowa, Iowa City, IA, USA). A cavity of the same dimensions, but deeper (approximately 8 mm), was similarly prepared in a poly(methyl methacrylate) block for the manufacture of inlays. Before taking an optical impression, the prepared cavity was coated with a thin layer of Vita CEREC Powder (CEREC Propellant, Vita Zahnfabrik, Bad Säckingen, Germany). A standardized inlay with a convex occlusal surface was designed and fabricated with a CEREC 3 CAD/CAM device (Sirona A.G., Bensheim, Germany). Twenty identical inlays were cut from commercial lithium disilicate glass–ceramic blocks (IPS e.max CAD for CEREC and inLab, HT A3/I12, Ivoclar Vivadent, Schaan, Liechtenstein; Batch # N32756). These inlays were crystallized in a furnace (VITA Vacumat 40T, Vita Zahnfabrik) according to the manufacturer’s instructions (10 min at 820°C and 7 min at 840°C).

2.2. Study design and bonding procedure (Fig. 1)

The prepared cavities and ceramic inlays were divided randomly into four experimental groups: two inlay luting procedures (dual-cure resin cement versus light-cure composite) were each performed by two operators in their final year of a specialized clinical training program (Master-after-Master in Restorative Dentistry).
All ceramic inlays were pretreated similarly. Immediately before bonding, hydrofluoric acid (IPS Ceramic Etching gel, Ivoclar Vivadent) was applied for 60 s to all cavity-facing surfaces, which were then rinsed thoroughly with water and air-dried. Monobond Plus (Ivoclar Vivadent) was applied and left undisturbed for 60 s and then air-dried. An unfilled adhesive resin (Heliobond, Ivoclar Vivadent) was applied but not light-cured to improve resin infiltration into the etched pits in the ceramic surface [2,9].

The inlays were seated in accordance with the manufacturer’s instructions (Table 1). After seating, final light-curing was applied to the occlusal surface for a single cycle of 40 s. The light had to travel 8 mm through the ceramic block before reaching the surface being investigated. Light-curing was performed with a high-power light-emitting diode (LED) light-curing unit (Bluephase 20i, Ivoclar Vivadent). Light intensity was controlled using a Bluephase meter (Ivoclar Vivadent) to ensure a light output of at least 2000 mW/cm² [10].

2.3. Micro-tensile bond strength testing

The restored cavities were stored in water for 1 week at 37°C, and were then sectioned perpendicular to the cavity bottom using a 300-µm diamond cut-off wheel (M1D10, Struers A/S, Ballerup, Denmark) mounted in an automatic precision
water-cooled cut-off machine (Accutom 50, Struers A/S) to obtain rectangular 1 × 1-mm non-trimmed micro-specimens for micro-tensile bond strength (μTBS) testing. The micro-specimens were kept moist until tested. They were attached to a notched BIOMAT jig [11] with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin, Ohtawara, Japan) and stressed at a crosshead speed of 1 mm/min until failure in an LRX testing device (Lloyd, Hampshire, UK) using a load cell of 100 N. After measuring the exact dimensions of each fractured beam with a digital caliper (CD-15 CPX, Mitutoyo, Tokyo, Japan), μTBS was measured (in MPa) by dividing the recorded force (in N) at the time of fracture by bond area (in mm²). If a specimen failed before testing, a bond strength of 0 MPa was used in statistical analyses. The number of pre-testing failures was also noted. The data were evaluated statistically with a two-way analysis of variance (ANOVA) and post hoc Tukey multiple comparison at a significance level of 0.05.

2.4. Failure analysis

The failure mode was determined at a 50 × magnification with a stereomicroscope (Wild M5A, Heerbrugg, Switzerland). Representative specimens in each group were selected for scanning electron microscopy (SEM) using standard specimen processing
techniques, including fixation in 2.5% glutaraldehyde in cacodylate buffer solution, dehydration in ascending concentrations of ethanol and chemical drying using hexamethyldisilazane (HMDS). After being mounted on stubs, the specimens were coated with a thin gold layer using an automatic sputter coater (JFC-1300, JEOL, Tokyo, Japan) and they were then subjected to SEM (JSM-6610 LV, JEOL).

3. Results

The µTBS values and results of the multiple comparisons are presented in Table 2 and Fig. 2. The two-way ANOVA revealed significant differences in bonding effectiveness between the two adhesive procedures ($P < 0.0001$).

The overall mean in the direct composite group ($33.3 \pm 12.0$ MPa) was almost six times higher than that in the luting cement group ($5.7 \pm 7.9$ MPa). Both operators performed equally ($P = 0.6550$), though not in every group, as a significant interaction effect was recorded ($P = 0.0013$). Both operators obtained significantly higher bond strength values with the direct composite compared with the dual-cure luting cement ($P < 0.0001$). Pre-testing failures were only observed in the dual-cure cement group, and 75% of the pre-testing failures were caused by a single operator (Table 2).
Distribution of the failure mode is summarized in Table 2, and representative SEM photomicrographs of the fractured surfaces after μTBS testing are shown in Figs 3 and 4. The type of failure varied considerably between the two procedures. With dual-cure cement, more failures occurred at the resin–dentin interface and exposed the hybrid layer over large parts of the fractured surface. The second predominant failure location was the adhesive resin, which contained numerous small (< 1 µm) bubbles in almost all specimens. A few specimens luted with dual-cure cement failed in the ceramic or at the resin–ceramic interface. Specimens luted with direct composite almost never fractured along the resin–dentin interface, and if the hybrid layer was exposed, it was only over a very small area of the fractured surface. In the direct composite group, most failures occurred within the resin composite, or at the resin–ceramic interface.

4. Discussion

The objective of this study was to compare the effectiveness of a direct light-cure composite with that of a conventional dual-cure composite cement for luting of CAD/CAM ceramic inlays in vitro. The statistical analysis revealed that, independently of the operator, the bonding effectiveness of the alternative luting method was
significantly higher than that of the conventional method. Therefore, the null-hypothesis tested was rejected.

IPS e.max CAD is categorized as a lithium disilicate glass–ceramic. This ceramic has high mechanical properties compared with conventional feldspathic or leucite-reinforced glass ceramics [12,13]. We aimed to determine whether even endo-crowns, a type of restoration where the crown and post are designed as a “monobloc”, could be cemented with light-cure materials only. Therefore, a highly translucent type of ceramic was chosen.

Variolink II luting cement is known for its high bonding effectiveness and superior durability [2,14], even when exposed to long-term thermocycling [7,15]. The bond strength values of Variolink II remained stable after thermocycling followed by storage in water for 6 months [7]. A low bonding effectiveness resulted, especially when compared with results obtained by the light-curing method. A previous study that evaluated µTBS to flat surfaces showed that the direct composite performed better, but Variolink yielded one of the best scores among conventional luting cements [16]. The µTBS procedure in that earlier study was very similar to what we used and was performed in the same laboratory. A number of possible explanations exist for the observed differences: (1) the different cavity design, resulting c-factor (cavity versus flat
surface; thick versus thin ceramic part) and reduced light transmission; (2) the different adhesive systems applied (two-step versus three-step etch and rinse); (3) the different ceramic (lithium disilicate glass–ceramic versus feldspathic ceramic); or (4) the different light-curing units (conventional quartz–tungsten–halogen lamp versus high-power LED lamp). Because the specimens failed predominantly at the resin–dentin interface (Table 2), the first two explanations appear to be the most plausible. When using Variolink II, it is crucial that the light passes through the entire length of the IPS e.max CAD for curing to occur. Earlier studies found that curing through 7-mm blocks reduced the bonding performance significantly [16,17]. Two-step etch and rinse adhesives are more technique sensitive, however, especially in more complex cavity configurations such as those used in this study [18,19].

Box-shaped Class-I cavities were prepared to make adhesion to the bottom of the cavity more difficult, thereby simulating clinical conditions. Water or resin tends to pool in the corners of such cavities, making it impossible to standardize surface moisture. Moreover, whereas the corners of the surface are too wet, the middle part of the surface may be too dry [20]. It is often very difficult to remove water or resin from such a narrow occlusal Class-I cavity, which may result in a pronounced water content in the adhesive resin. This excess water cannot mix with the adhesive primer. When
ethanol is evaporated by air drying, water droplets are formed in the adhesive resin [21,22]. This was confirmed by the numerous small droplets observed by SEM in almost all specimens in this group (Fig. 3d). Obviously this water excess does not contribute to interfacial strength or stability [23]. Factors such as dentin moisture [24], seating pressure [25,26] and even total application time [27] may differ between operators, which may also explain the observed technique sensitivity when using conventional dual-cure cement.

In contrast, when both operators used the alternative method (direct composite and a two-step self-etch adhesive), significantly higher bond strength values were obtained and no operator effect was observed. Since no chemical initiator is included in Clearfil SE Bond or Clearfil AP-X, polymerization relies completely on the light transmitted through the 8-mm ceramic, which corresponds approximately to the thickness of a so-called endo-crown. To maximize light transmission, a powerful LED curing device with an output of more than 2000 mW/cm² was used. To mimic clinical conditions, the occlusal surface was not flat and allowing for additional light scattering. A thicker ceramic is known to reduce light intensity and other factors such as the constitution of the ceramic (leucite-reinforced versus lithium–disilicate glass–ceramic) and opacity [28-30]. This reduced light intensity affects the degree of conversion
[31,32], curing depth and hardness [32,33].

Polymerization was performed through a thick ceramic layer. Therefore, apart from a higher light intensity, an extended polymerization time of 40 s was also chosen. With lithium–disilicate ceramic, in particular, a reduction in curing time and increase in ceramic thickness can result in insufficient polymerization [28].

Limited research is available regarding the use of direct light-cure composites to lute ceramic inlays. However, some in vivo studies compared both techniques with a follow-up period of up to 12 years of clinical service [34-36]. One clinical study that investigated Class-II ceramic restorations after 4 years found that a significant operator effect resulted, but there was no influence from the luting material used (light-cure ormocer versus high-viscosity dual-cure resin cement) [34]. Failure rates of 8% and 16% were obtained after 8 and 12 years, respectively, in other in vivo examinations that compared three different types of cement and one direct composite for luting IPS Empress inlays [35,36]. After 4 years, a significantly greater excess of luting composite was present when cement was used (Variolink Low; Ivoclar Vivadent) compared with the direct composite (Tetric; Ivoclar Vivadent). This indicates that high-viscosity materials are superior for use in daily clinical practice [35]. In addition to the high µTBS values obtained, one earlier report noted that the amount of bulk fractures was
significantly higher when the light-curing material was used than that for the low viscosity resin cement over a 12-year clinical observation period [34]. We believe that this is related to insufficient curing of the light-cure composite, which appeared less important in this study, given the higher bond strengths in the direct composite group. Recent innovations in light-curing units should also be taken into account; conventional and relatively inefficient halogen units have been replaced by high-power, highly efficient LED devices, as used in this study [37].

Besides the improved bond effectiveness observed in this in vitro study, the use of a direct composite to lute ceramic inlays yields several clinical advantages. Polymerization of the interface is instantaneous and on-demand because no chemical curing is required. This results in extended working/seating time and facilitates the luting of this type of restoration. If a light-cure composite is used with a self-etch adhesive, the application procedure may be less technique-sensitive and therefore less prone to application errors. It is also easier to remove composite excess when a viscous direct composite resin rather than a low viscous material is used.

The higher viscosity of direct composites compared with conventional luting cement may also be affected by pre-heating [38-40] or ultrasonic application [41] and can therefore be adjusted based on clinical demand.
5. Conclusions

The *in vitro* findings suggest that high-power LED light-curing (more than 2000 mW/cm²) of a direct composite offers a viable alternative to luting lithium disilicate glass–ceramic CAD/CAM restorations in Class-1 cavities. Higher bond strength values were obtained when the direct composite was light-cured. Furthermore, the luting procedure was less technique-sensitive and clinically more appealing.

Author Disclosure Statement

The authors have no financial affiliation to any of the companies whose products are included in this article.
References


Figure Captions

Fig. 1 – Schematic of study set-up. (a) Standardized box-shaped Class-I cavity was prepared at occlusal surface. (b) CAD/CAM-fabricated ceramic inlays were luted by light-curing or dual-curing. (c) Resin–dentin micro-specimens (1 mm × 1 mm) were cut with automated diamond saw and (d) stressed until failure.

Fig. 2 - Box whisker plots of µTBS (MPa). Groups showing no statistical difference are connected by a horizontal line (Tukey multiple comparison, P > 0.05).

Fig. 3 - (a) SEM photomicrograph of fractured dentin-side surface in Group 1 (luting composite, Operator A) showing mixed failure pattern. Circular scratches at cavity bottom resulted from contact with tip of diamond bur during cavity preparation. (b) Higher magnification of specimen in Fig. 3a; failure occurred in hybrid layer and adhesive resin. Numerous droplets visible in adhesive resin. (c) Overview SEM photomicrograph of fractured ceramic-side surface in Group 3 (luting composite, Operator B). In contrast with Fig. 3a, failure occurred almost completely in adhesive resin. (d) Higher magnification of specimen in Fig. 3c; typical accumulation of small droplets (smaller than 500 nm) over entire surface. White rectangle shows origin of detail presented (d).

Fig. 4 - (a) SEM photomicrograph of fractured dentin-side surface in Group 2 (direct
composite, Operator A) showing mixed failure pattern. Failure occurred within most of the composite and at the composite/ceramic interface. (b) Higher magnification of specimen in Fig. 4a, intersectional area with failure at level of hybrid layer and adhesive resin. (c) SEM photomicrograph of fractured ceramic-side surface in Group 4 (direct composite, Operator B). Failure occurred over most of the adhesive interface and in the adhesive resin (d) Higher magnification of specimen in Fig. 4c; intersectional area with failures between adhesive resin and adhesive interface. In contrast with Groups 1 and 3 (luting composite), no droplets were detected, and dentin surface was covered with smear layer. White rectangle shows origin of detail presented (d).
Fig. 3

Luting Composite

Operator A

a  W550m  5540  X550x

Operator B

b  W550m  5540  X550x

c  W550m  5540  X550x

d  W550m  5540  X550x

interface adhesive resin

adhesive resin
Fig. 4

Direct Composite

Operator A

Operator B
### Table 1 – Materials tested and application protocol

<table>
<thead>
<tr>
<th>Group</th>
<th>Product name</th>
<th>Components</th>
<th>Main ingredients</th>
<th>Application protocol</th>
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<tr>
<td>Luting composite</td>
<td>ExciTE F DSC / Variolink II</td>
<td>Total Etch [N05612]</td>
<td>37% phosphoric acid</td>
<td>Etch cavity wall with Total Etch for 15 s and rinse; apply ExciTE F DSC with a brush, on enamel and dentin of cavity wall, and agitate for at least 10 s; dry for 1–3 s; light-cure for 20 s; mix equal amounts of base and catalyst for 10 s; apply the mixture into cavity with a brush; insert inlay and remove excess cement; light-cure for 40 s.</td>
</tr>
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<td>(Ivoclar Vivadent, Schaan, Liechtenstein)</td>
<td>ExciTE F DSC [N24042]</td>
<td>Phosphonic acid acrylate, dimethacrylate, HEMA, highly dispersed silicon dioxide, ethanol, catalysts, stabilizers, fluoride, initiators</td>
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<td></td>
<td>Base paste [N23694]</td>
<td>Bis-GMA, UDMA, TEGDMA, inorganic filler, ytterbium trifluoride, initiators, stabilizers</td>
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<td></td>
<td>Catalyst [N22039]</td>
<td>Bis-GMA, UDMA, TEGDMA, inorganic filler, ytterbium trifluoride, benzoyl peroxide, stabilizers</td>
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<td>Direct composite</td>
<td>Clearfil SE Bond / Clearfil AP-X</td>
<td>Primer [00903B]</td>
<td>MDP, HEMA, hydrophilic dimethacrylate, photoinitiator (CQ, DEPT, others), water</td>
<td>Apply primer and leave it in place for 20 s; gently air dry; apply bond, thin with air pressure; light cure for 10 s; apply Clearfil AP-X and insert inlay; remove excess paste; light-cure for 40 s.</td>
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<td>(Kuraray Medical, Kurashiki, Japan)</td>
<td>Bond [01332A]</td>
<td>MDP, bis-GMA, HEMA, hydrophilic dimethacrylate, microfiller</td>
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<td>Clearfil AP-X [000388]</td>
<td>Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, CQ, catalysts, accelerators, pigments, others</td>
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Table 2 – μTBS and failure analysis

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<tr>
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<th>Mean ± S.D. (MPa)</th>
<th>PTF/n</th>
<th>Dentin</th>
<th>Interface</th>
<th>Composite</th>
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<th>Ceramic</th>
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<td>Luting composite – Operator A</td>
<td>9.0 ± 8.6$^b$</td>
<td>7/21</td>
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<td>45</td>
<td>30</td>
<td>9</td>
<td>15</td>
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<tr>
<td>Luting composite – Operator B</td>
<td>1.0 ± 4.2$^c$</td>
<td>19/20</td>
<td>0</td>
<td>100</td>
<td>0</td>
<td>0</td>
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<tr>
<td>Direct composite – Operator A</td>
<td>29.7 ± 8.3$^a$</td>
<td>0/17</td>
<td>0</td>
<td>14</td>
<td>37</td>
<td>49</td>
<td>0</td>
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<tr>
<td>Direct composite – Operator B</td>
<td>35.8 ± 13.6$^a$</td>
<td>0/26</td>
<td>2</td>
<td>9</td>
<td>70</td>
<td>19</td>
<td>0</td>
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* Pre-testing failures were excluded from failure analysis.

$\mu$TBS: micro-tensile bond strength, PTF: number of pre-testing failures, $n$: total number of specimens, S.D.: standard deviation.

Means with same superscript letter were not significantly different ($P > 0.05$, post hoc Tukey multiple comparison test)