Cementation in adhesive dentistry: the weakest link

Jongsma, L.A.

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CHAPTER 6

Shear bond strength of three dual-cured resin cements to dentin analyzed by finite element analysis

This chapter is submitted to Dental Materials: Leontine A. Jongsma, Niek de Jager Ir., Cornelis J. Kleverlaan, Prem Pallav†, Albert J. Feilzer. Shear bond strength of three dual-cured resin cements to dentin analyzed by finite element analysis
Abstract

Objectives: To determine the shear bond strength to bovine dentin of dual-cured resin cements cured in different circumstances, the contraction stress and volumetric shrinkage in both polymerization modes, and to review the failure stress distribution at the cement-tooth interface with finite element analysis.

Methods: The volumetric shrinkage of the cements RelyX Unicem, Panavia F 2.0 and DC Core Automix was determined by mercury dilatometry. Polymerization contraction stress was determined using a constraint tensilometer set-up. For the shear bond strength test, cement discs on bovine root dentin (self-cured and dual-cured), composite discs cemented to dentin (self-cured and dual-cured), and dentin cemented to dentin (self-cured) specimens were fabricated. Specimens were stored in water for 24 h (37 °C, 100% humidity) and tested (crosshead speed 1 mm/min). FE modeling of the specimens was carried out in order to calculate the maximum shear stresses in the cement-dentin interface. Differences between groups were determined using two-way ANOVA with Tukey post hoc tests, and paired samples $t$-tests ($\alpha < 0.05$).

Results: Panavia F 2.0 showed significantly lower volumetric shrinkage than the other cements. Dual-curing lead to higher contraction stresses for all tested cements compared to self-curing. RelyX Unicem showed higher volumetric shrinkage when dual-cured. Shear bond strength and maximum shear stress was positively influenced by dual-curing. DC Core Automix performed best and Panavia F 2.0 worst in terms of shear bond strength and maximum shear stress.

Significance: Dual-curing the cements leads to higher contraction stress, and also to higher shear bond strengths and maximum shear stresses in the case of RelyX Unicem.
Introduction

The retention and clinical success of indirect restorations relies heavily on the bond strength the cement will achieve and maintain to the substrates it is bonded to [1]. The cements that are most used today are dual-cured resin cements. Its bond quality may depend on many variables, like the mode of polymerization [2-4]. An important advantage of dual-cured cements is their ability to be light-cured to shorten treatment time, whilst a proper cure is secured in places where the light is not likely to reach the cement, for instance beneath an inlay, opaque crown materials like zirconium-dioxide reinforced ceramic crowns, or in deeper aspects of a root canal or post space. Nevertheless, previous research shows that sufficient curing in these places is not always obvious. Contradictory results are published. On one hand, light-curing is observed to be less effective in deep aspects of post spaces, resulting in a lower degree of conversion [5, 6] which might be compensated for by the dual-cure mode as is reported by Pereira who did not find differences in degree of conversion for a dual-cured resin cement in different root canal regions [7]. On the other hand, lower cohesive strength [8], and depending on the cement, a lower diametral tensile strength [9] within apical parts of the post space, even for dual-cured cements were reported. The light-transmitting ability of the post [6] or restorative material [10] plays a role together with the distance from the light source [5].

An important problem with resin cements is their volumetric shrinkage. This is caused by the cross-linking of monomers to polymer chains which results in volume loss of the cement. In an unconstraint situation this volume loss is compensated for by viscous flow of the cement [11]. In a constraint condition however, when the cement is adhesively bonded to one or more walls, the flow of the cement is hindered, and the volume loss of the cement results in internal stresses known as contraction stress [12, 13]. The amount of stress can be predicted by calculating the Configuration factor or C-factor which has been described by Feilzer et al. as the ratio between bonded and unbonded surface of a cavity [14]. With a high C-factor, the flow of the resin cement to compensate for volume loss as a result of polymerization shrinkage of the cement is very limited. Therefore, high stresses are generated. This can negatively influence the bond strength between cement and dentin, and even disrupt this bond. The higher the C-factor, the greater this problem will be [14-17], although this effect can be counteracted by the compliance of the cavity walls [13, 18]. Clinically, these effects
are encountered with the cementation of resin bonded (indirect) restorations such as (prefabricated fiber) posts, (partial) crowns, inlays and onlays. The C-factor inside post space preparations has been calculated by Bouillaguet [16] and Jongsm [19] and has an estimated value of 200-220. Therefore, cementing a post into a root canal space leading to a reliable result with predictable quality is not an easy task. This is illustrated by the fact that the most common mode of failure encountered with fiber-reinforced composite post restorations clinically, is debonding of the post [20].

However, the contraction stresses involved in the cementation of an indirect restoration are not solely a result of the C-factor. The mode of polymerization also plays an important role. Light curing a resin cement leads to a fast polymerization process, limiting the time for viscous flow of the cement, and thereby increasing the contraction stresses [21]. Self-curing of a dual-cure cement, however, often leads to lower degrees of cure [22, 23] and therefore lower mechanical properties [24-26]. Also, it has been shown that thin resin layers have higher tensile strengths compared to thicker resin layers [27]. This is likely to be of influence.

The aim of the study was to gain a better understanding of the variables curing mode, volumetric shrinkage, polymerization contraction and C-factor on the shear bond strength to bovine dentin and the maximum shear stresses in the cement-dentin interface as calculated with Finite Element Analysis (FEA).

**Materials and methods**

In this study, the shear bond strength of the cements RelyX Unicem, Panavia F 2.0, and DC Core Automix (Table 6.1) to bovine dentin was determined under different circumstances. The cements were either self- or dual-cured and placed in bulk, as a cement layer underneath a composite disc, or in between two bovine root dentin layers (Table 6.2, Figure 6.1). The specimens were prepared by removing the crowns of bovine teeth leaving the roots which were ground flat and embedded in resin (PMMA Self Curing Clear, Vertex, NL) to create dentin discs.
<table>
<thead>
<tr>
<th>Product</th>
<th>Manufacturer</th>
<th>Batch number</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil DC Core Automix Paste</td>
<td>Kuraray</td>
<td>0123AA</td>
<td>Catalyst paste: Bis-GMA, TEGDMA, silanated colloidal silica, barium glass, D,L-camphorquinone, benzoyl peroxide Universal paste: Bis-GMA, TEGDMA, silanated colloidal silica, barium glass, N,N-diethanol p-toluidine</td>
</tr>
<tr>
<td>Clearfil SA Primer</td>
<td>Kuraray</td>
<td>0066CA</td>
<td>Salicylic acid monomer</td>
</tr>
<tr>
<td>Clearfil Photo Bond Bonding Agent Universal</td>
<td>Kuraray</td>
<td>0536AA</td>
<td>N,N-diethanol p-toluidine, sodium benzene sulfinate, ethanol</td>
</tr>
<tr>
<td>Clearfil Photo Bond Bonding Agent Catalyst</td>
<td>Kuraray</td>
<td>0439AA</td>
<td>MDP, Bis-GMA, HEMA, hydrophobic aliphatic dimethacrylate, camphorquinone, benzoyl peroxide Powder: glass fillers, silica, calcium hydroxide, self-cure initiators, pigments, light-cure initiators. Liquid: methacrylated phosphoric esters, dimethacrylates, acetate, stabilisers, self-cure initiators</td>
</tr>
<tr>
<td>Panavia F 2.0 Paste A</td>
<td>Kuraray</td>
<td>0431AB</td>
<td>Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, sodium aromatic sulfinate (TPBSS), N, N-diethanol-p-toluidine, surface treated (functionalized) sodium fluoride, silanated barium glass</td>
</tr>
<tr>
<td>Panavia F 2.0 Paste B</td>
<td>Kuraray</td>
<td>0220AB</td>
<td>MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated silica, photoinitiator, dibenzoylperoxide</td>
</tr>
<tr>
<td>Panavia F 2.0 ED Primer II Liquid A</td>
<td>Kuraray</td>
<td>0280CA</td>
<td>HEMA, MDP, 5-NMSA, water, accelerator</td>
</tr>
<tr>
<td>Panavia F 2.0 ED Primer II Liquid B</td>
<td>Kuraray</td>
<td>0156AA</td>
<td>5-NMSA, accelerator, water, sodium benzene sulfinate</td>
</tr>
<tr>
<td>Panavia F 2.0 Oxyguard II Filtek Supreme XT restorative composite</td>
<td>3M ESPE</td>
<td>Multiple</td>
<td>Polyethylene glycol, glycerine, sodium-benzenesulfinate, N,N-diethanol p-toluidine Non-agglomerated/non-aggregated 75nm silica nanofiller, non-agglomerated/non-aggregated 15-20nm zirconia nanofiller, loosely bound agglomerated zirconia/silica nanocluster consisting of 5-20nm primary zirconia/silica particles, Bis-GMA, TEGDMA, Bis-EMA</td>
</tr>
</tbody>
</table>

Kuraray medical Inc., Okoyama, Japan, 3M ESPE, Seefeld, Germany, RTD, St. Egrève France. Bis-GMA: Bisphenol A diglycidylmethacrylate; TEGDMA: Triethylene glycol dimethacrylate MDP: 10-methacryloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; 5 NMSA: N-methacryloxy-5-aminosalicylic acid.
The discs were polished wet with 600 grit sanding paper (silicon grinding paper, Carbimet, Buehler, USA) to obtain a standardized smear layer, and stored at room temperature at 100% humidity. All cements were applied according to the manufacturers’ instructions, and in the case of the dual-cured groups, light-cured with a Elipar Freelight (3M Espe, Seefeld, Germany). Each cement was tested with each test set-up (n = 10). In the DC-SC and DC-DC groups, the cements were applied in a rubber mold with a diameter of 5 mm and a height of 1 mm. Before curing of the cement, the cement was covered with a plastic matrix strip. In the DCC-SC and DCC-DC groups, a small amount of cement was applied and a composite (Filtek Supreme XT, 3M-ESPE, Seefeld, Germany) disc with a diameter of 5 mm and a height of 1 mm was cemented to the dentin. In the DCD-SC groups, two dentin discs were cemented together. In order to obtain a standardized bonding surface, a perforated (5 mm diameter) transparent matrix band was applied to the surface of one of the dentin discs as they were cemented together.

<table>
<thead>
<tr>
<th>Table 6.2</th>
<th>Description of the test set-ups in the different research groups.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Group</strong></td>
<td><strong>Specimen</strong></td>
</tr>
<tr>
<td>DC-SC</td>
<td>Dentin – Cement</td>
</tr>
<tr>
<td>DC -DC</td>
<td>Dentin – Cement</td>
</tr>
<tr>
<td>DCC-SC</td>
<td>Dentin – Cement – Composite</td>
</tr>
<tr>
<td>DCC-DC</td>
<td>Dentin – Cement – Composite</td>
</tr>
<tr>
<td>DCD-SC</td>
<td>Dentin – Cement – Dentin</td>
</tr>
</tbody>
</table>

All specimens were stored at 37 °C for 24 h in the dark before testing. After this, the rubber molds were removed from the specimens, and the specimens were placed in the testing device for shear testing until failure. The testing was performed with the Instron 6022 Tensilometer (Instron ltd., Wycombe, UK) at a crosshead speed of 1 mm·min⁻¹. The load at failure was recorded, and the shear strength was calculated. The mode of failure, *i.e.* adhesive, cohesive or mixed, was determined by visual inspection.
In addition to the shear bond strength measurements, the volumetric shrinkage and polymerization contraction stress were determined for the same cements. Volumetric shrinkage measurements were performed with the use of a mercury dilatometer at 23 ± 0.1°C as reported by de Gee et al. [28]. The specimens (n = 6) were either dual-cured using an Elipar Trilight (3M ESPE, Seefeld, Germany) for 40 s in a standard mode, or left to self-cure. The measuring continued for 30 min.

Polymerization contraction stress measurements were carried out using a constraint tensilometer set-up as described by Kleverlaan et al. [29]. The specimens (n = 6) were either light-cured (3M ESPE Elipar Trilight) for 40 s or self-cured. During a period of 30 min, the contraction stress development was measured, while the distance between the glass and steel bolt head was kept constant through counteracting feedback displacement of the cross-head. This simulated a restoration in a fully rigid situation, where the cavity walls do not yield to the contraction forces.
Finite element analysis

Three-dimensional FE models of the three different test specimens DC, DCC, and DCD were created. The finite element modeling was carried out using FEMAP software (FEMAP 10.1.1; Siemens PLM software, Plano, Texas, USA), while the analysis was done with NX Nastran software (NX Nastran; Siemens PLM Software, Plano, Texas, USA). The models were designed according to the dimensions of the test specimens of the shear bond strength test. The cement layer was designed with a thickness of 50 μm. Since the models were symmetrical in geometry they were split in half specimens, with the nodes in the centric plane allowing for sliding in the surface only. The models were composed of 22,508 to 39,491 parabolic tetrahedron solid elements. For the resin cements an elastic modulus of 7.5 GPa [26, 30, 31] was used. For the resin composite material and the dentin, values of 10.5 GPa [32] and 18.3 GPa [33] were used, respectively. The Poisson’s ratio was 0.3 in all cases. The anisotropy of the dentin was not taken into account.

The experimentally determined loads from the shear bond strength tests were applied on the nodes at the outside central area of the dentin disc. In order to simulate the placement and the support in the test set-up of the specimen, the nodes in the area, where the force was applied and the nodes at the outside plane of the loaded dentine disc were fixed with no movement in the direction perpendicular to the force and the nodes of the elements at the opposite outside central area of the other disc were fixed with no movement in any direction (Figure 6.2).

The analysis was carried out with the load as primary input followed by a second calculation taking into account the shrinkage of the cement layer. The obtained values of the volumetric shrinkage and the polymerization contraction stresses (Table 6.4) were used. In an FE model simulating the used tensilometer set-up, the contraction stresses ($\sigma_{\text{FEA}}$) were calculated using the values of the shrinkage determined by the dilatometer. As described by Ausiello et al. [34], the contraction stresses found in the test set-up ($\sigma_{\text{TEST}}$) were considered to be caused by the constraint portion of the shrinkage, the remaining shrinkage being free shrinkage. The constraint shrinkage was calculated as the $\sigma_{\text{TEST}} / \sigma_{\text{FEA}}$ portion of the total shrinkage. This calculated constraint shrinkage was used as shrinkage to calculate the stresses due to the shrinkage of the cement layer in the models of the test specimens. Hereafter, the combined stresses of the stresses due to the loads and the shrinkage of the cement...
layer were calculated using the linear combination facility of FEMAP. The maximum shear stresses at the cement-dentin interface in the horizontal plane were reported.

**Figure 6.2** FE model of the DCC-DC specimen, with the maximum shear stresses in the horizontal plane in the cement-dentin interface of DC Core Automix.

**Statistical analysis**
Two-way ANOVA and Tukey’s post-hoc tests (SPSS 15.0, SPSS inc., Chicago, Mn, USA) were used to analyze the 30 minute results of the volumetric shrinkage and contraction stress tests, as well as the differences between groups of the shear bond strength test, and the maximum shear stresses as obtained with FEA. The volumetric shrinkage and contraction stress data measured at 5, 15, and 30 minutes, respectively,
were analyzed with paired $t$-tests. A P-value < 0.05 was considered statistically significant.

**Results**

The results of the shear bond strength of the different cements to bovine dentin are summarized in Table 6.3A. Two-way Analysis of Variance showed a significant effect on cement ($F = 14.3; P < 0.001$) and method ($F = 9.9; P < 0.001$). There was also a significant interaction between cement and method ($F = 2.6; P = 0.011$). When looking at the mean overall results of the cements without considering the method used, all cements proved to be significantly different from each other. Panavia F 2.0 presented the lowest and DC Core Automix the highest shear bond strength values. The Tukey post-hoc tests were used to identify individual significant differences within the cements and methods and are also summarized in Table 6.3A.

The modes of failure are summarized in Table 6.3B. Most failures were adhesive failures between cement and dentin, but a small portion of mixed cohesive-adhesive failures were observed. Pure cohesive failures were not observed in this study.

The volumetric shrinkage and polymerization contraction of the different cements are summarized in Table 6.4 and graphically depicted in Figure 6.3. Two-way Analysis of Variance of the volumetric shrinkage at 30 min showed a significant effect on cement ($F = 22.2; P < 0.001$) and curing mode ($F = 7.0; P = 0.0013$). There was no significant interaction between cement and curing mode. Tukey post-hoc tests (Table 6.4) showed that overall Panavia had a significantly lower volumetric shrinkage than the other two cements, which were not statistically different from each other. RelyX Unicem showed a higher volumetric shrinkage after dual-curing compared to after dual-curing after 30 min, which was not the case for the other cements at a statistically significant level.
Table 6.3  (A) Mean shear strength in MPa (with their standard deviation in parentheses) of the different methods and cements and their overall results, as obtained with shear bond strength test. (B) Percentage of adhesive failure of the different methods and cements. (C) Maximum shear stresses in MPa (with their standard deviation in parentheses) in the cement-dentin interface in the horizontal plane, as calculated with FEA.

<table>
<thead>
<tr>
<th></th>
<th>RelyX Unicem</th>
<th>Panavia F 2.0</th>
<th>DC Core Automix</th>
</tr>
</thead>
<tbody>
<tr>
<td>DC-SC</td>
<td>5.7 (3.4)&lt;sup&gt;AB&lt;/sup&gt;</td>
<td>6.4 (2.6)</td>
<td>7.9 (1.9)&lt;sup&gt;ABC&lt;/sup&gt;</td>
</tr>
<tr>
<td>DC-DC</td>
<td>10.8 (3.7)&lt;sup&gt;A&lt;/sup&gt;</td>
<td>7.1 (1.5)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>13.0 (3.1)&lt;sup&gt;Ab&lt;/sup&gt;</td>
</tr>
<tr>
<td>DCC-SC</td>
<td>7.6 (1.7)&lt;sup&gt;C&lt;/sup&gt;</td>
<td>9.3 (3.0)</td>
<td>10.8 (2.4)</td>
</tr>
<tr>
<td>DCC-DC</td>
<td>15.4 (8.2)&lt;sup&gt;B,C,Da&lt;/sup&gt;</td>
<td>9.3 (3.8)&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>13.4 (6.1)&lt;sup&gt;Bb&lt;/sup&gt;</td>
</tr>
<tr>
<td>DCD-SC</td>
<td>9.9 (4.2)&lt;sup&gt;Da&lt;/sup&gt;</td>
<td>7.2 (1.7)&lt;sup&gt;b&lt;/sup&gt;</td>
<td>14.4 (3.5)&lt;sup&gt;Cab&lt;/sup&gt;</td>
</tr>
<tr>
<td>Overall cement</td>
<td>9.8 (5.6)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>7.9 (2.8)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>11.9 (4.3)&lt;sup&gt;n&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
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<th>Rely X Unicem</th>
<th>Panavia F 2.0</th>
<th>DC Core Automix</th>
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<tr>
<td>DC-SC</td>
<td>90</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>DC-DC</td>
<td>60</td>
<td>90</td>
<td>80</td>
</tr>
<tr>
<td>DCC-SC</td>
<td>90</td>
<td>100</td>
<td>80</td>
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<td>DCC-DC</td>
<td>70</td>
<td>100</td>
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<tr>
<td>DCD-SC</td>
<td>100</td>
<td>70</td>
<td>100</td>
</tr>
<tr>
<td>Overall cement</td>
<td>82</td>
<td>92</td>
<td>90</td>
</tr>
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</table>

<table>
<thead>
<tr>
<th></th>
<th>RelyX Unicem</th>
<th>Panavia F 2.0</th>
<th>DC Core Automix</th>
</tr>
</thead>
<tbody>
<tr>
<td>DC-SC</td>
<td>14.5 (8.6)&lt;sup&gt;AB&lt;/sup&gt;</td>
<td>16.5 (5.8)</td>
<td>20.0 (4.9)</td>
</tr>
<tr>
<td>DC-DC</td>
<td>28.0 (9.5)&lt;sup&gt;AC,Da&lt;/sup&gt;</td>
<td>19.0 (4.1)&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>28.0 (6.7)&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>DCC-SC</td>
<td>16.0 (3.5)&lt;sup&gt;CE&lt;/sup&gt;</td>
<td>17.5 (5.7)</td>
<td>20.0 (4.5)</td>
</tr>
<tr>
<td>DCC-DC</td>
<td>28.5 (15.2)&lt;sup&gt;B,E,Fa&lt;/sup&gt;</td>
<td>18.5 (7.6)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>26.0 (11.9)</td>
</tr>
<tr>
<td>DCD-SC</td>
<td>15.0 (6.3)&lt;sup&gt;DF&lt;/sup&gt;</td>
<td>11.5 (2.8)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>22.0 (5.4)&lt;sup&gt;n&lt;/sup&gt;</td>
</tr>
<tr>
<td>Overall cement</td>
<td>20.4 (11.2)&lt;sup&gt;a&lt;/sup&gt;</td>
<td>16.6 (5.9)&lt;sup&gt;ab&lt;/sup&gt;</td>
<td>23.2 (7.6)&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>A,B,C,D,E,F</sup>: Statistically significant difference within the cement between the methods (columns).
<sup>a,b,c</sup>: Statistically significant difference within the method between the cements (rows).

Two-way Analysis of Variance of the polymerization contraction stress at 30 minutes showed a significant effect on cement (F = 12.3; P < 0.001) and curing mode (F = 1630.7; P < 0.001). There was no significant interaction between cement and
curing mode. Tukey post-hoc tests showed that overall, Panavia F 2.0 had significantly lower contraction stress values compared to RelyX Unicem and DC Core Automix, which were not statistically different from each other. Dual curing resulted in significantly higher contraction stresses for all three cements tested. Paired samples $t$-tests showed that there was a significant increase for the polymerization contraction stress at all time intervals ($5 \text{ vs. } 15$, $5 \text{ vs. } 30$, and $15 \text{ vs. } 30 \text{ min}$) for all cements tested.

**Table 6.4** Shrinkage (vol. %) and contraction stress (MPa) of the resin cements with their standard deviation in parentheses.

<table>
<thead>
<tr>
<th></th>
<th>Rely X Unicem</th>
<th>Panavia F 2.0</th>
<th>DC Core Automix</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Self-Cure</td>
<td>Dual-Cure</td>
<td>Self-Cure</td>
</tr>
<tr>
<td>Shrinkage (Vol. %)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 min</td>
<td>1.45 (0.11)</td>
<td>2.71 (0.39)</td>
<td>0.50 (0.33)</td>
</tr>
<tr>
<td>15 min</td>
<td>2.39 (0.19)</td>
<td>3.08 (0.38)</td>
<td>1.51 (0.38)</td>
</tr>
<tr>
<td>30 min</td>
<td>2.96 (0.19)$^a$</td>
<td>3.33 (0.39)$^b$</td>
<td>2.51 (0.29)$^i$</td>
</tr>
<tr>
<td>Contraction stress (MPa)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>5 min</td>
<td>0.03 (0.03)</td>
<td>16.25 (1.76)</td>
<td>0.02 (0.12)$^*$</td>
</tr>
<tr>
<td>15 min</td>
<td>1.20 (0.14)</td>
<td>18.00 (1.88)</td>
<td>0.11 (0.20)$^*$</td>
</tr>
<tr>
<td>30 min</td>
<td>2.56 (0.24)$^a$</td>
<td>19.08 (1.94)$^b$</td>
<td>1.62 (0.29)$^a$</td>
</tr>
</tbody>
</table>

*: No statistically significant difference between different time groups within the same cement and method (result of paired samples $t$-test)

$^{1,2}$: No statistically significant difference between curing modes within the same cements (result of two-way ANOVA).

$^a, b$: No statistically significant difference between cements within the same curing modes (result of two-way ANOVA).
Figure 6.3  (A) Mean volumetric shrinkage (%) as a function of time (min) of the different cements in different curing modes. (B) Mean contraction stress (MPa) as a function of time (min) of the different cements in different curing modes.
Finite element analysis

The experimentally determined loads from the shear bond strength tests and the results of the volumetric shrinkage and polymerization contraction stress were used to calculate the maximum shear stresses at the cement-dentin interface. These calculations were executed for the individual specimens enabling statistical analysis on these groups, similar to the analysis of the shear bond strength itself. These results are summarized in Table 6.3C. Two-way Analysis of Variance showed a significant effect on cement ($F = 9.6; P < 0.001$) and method ($F = 9.5; P < 0.001$). There was no significant interaction between cement and method, however. When considering the mean overall results of the cements, Panavia F 2.0 showed lower maximum shear stresses at failure compared to the other two cements. Within RelyX Unicem and DC Core Automix, the self-cured groups showed lower maximum shear stresses compared to the dual-cured groups. Although in the other two cements the same trend could be observed, there were no statistically significant differences between the groups.

In the DC-SC, DCC-SC groups, no statistically significant differences existed between cements. In the DC-DC group, Panavia F 2.0 showed lower shear stresses compared to RelyX Unicem and DC Core Automix, which did not differ from each other ($P < 0.05$). In the DCC-DC group, RelyX Unicem showed higher shear stress values compared to Panavia F 2.0 at a statistically significant level. In the DCD-SC group, DC Core Automix showed significantly higher shear stresses compared to Panavia F 2.0. There were no further statistically significant differences.

The highest stresses at the interface for the DC and DCC specimens are located near the stress application point, while the stresses in the DCD specimens are distributed much more evenly over the cement-dentin interface. This is caused by the larger distance from the point of stress application to the start of the cement-dentin interface in the DCD specimens.

Discussion

The shear bond strength values obtained in this study are comparable with other results from literature [35]. However, it should be noted that the variation of reported bond strength values is enormous, not only due to the different materials tested, but also because of the different testing methodologies, which can have a dramatic effect on
bond strength value measurements [36-38]. Moreover, the standard deviations are high, which is common with bond strength studies. Especially for shear bond strength tests, non-uniform stresses are generated within the specimens, which can have a significant effect on the mode of failure [36]. The results of shear bond strength testing are therefore not directly relatable to for example micro tensile bond strength tests or to the clinical situation in the human oral cavity.

To overcome this problem, finite element analysis was used to reveal the interfacial maximum stresses during shear stress measurements at the cement-dentin interface. To create a reliable finite element analysis model, the volumetric shrinkage and polymerization contraction stress of the cements are necessary to evaluate the additional stress induced by the curing of the cements themselves. Since there are no comparable data available in literature, the volumetric shrinkage and polymerization contraction stress were experimentally determined. The volumetric shrinkage of around 3% is comparable to the values measured for conventional composite restorative materials [29], although a higher volumetric shrinkage of 5.8% has been reported for DC Core Automix tested with a different testing method [39].

The maximum shear stresses in the cement-dentin interface and the maximum principal stresses in the cement layer were calculated. Since the maximum principal stresses in the cement layer were not higher than the cohesive strength of the cements (unpublished data), cohesive failure of the specimens was unlikely. This was supported by the observation that the majority of the shear bond test specimens failed adhesively, with only a small fraction of mixed failures (Table 6.3B). Therefore, the maximum shear stresses in the cement-dentin interface were used in this study. The values for the shear stresses in the horizontal plane, as obtained with FEA, are comparable to the reported values for microtensile bond strength to root dentin of DC Core Automix [40], which confirmed the validity of the FE models.

DC Core Automix showed the highest shear bond strengths of all three cements tested. The FEA results showed that the shear stresses of Panavia F 2.0 were the lowest values, but Rely X Unicem and DC Core Automix performed similarly. It is well established that a classical three-step etch and rinse system often provides higher bond strengths compared to self-etch and self-adhesive cements. Hayashi et al. stated that wet bonding of root canal dentin in general leads to higher shear bond strengths than using a self-etching system [41]. Viotti et al. reported higher microtensile bond strength values for one and two-step adhesive systems compared to self-adhesive
cements [42]. Panavia F 2.0 performed weakest in terms of shear bond strength and maximum shear strength. Perhaps the mild self-etching effect of ED primer was not enough to remove the smear layer in a sufficient way for a strong bond to be achieved. Apparently the self-adhesive properties of RelyX Unicem were sufficient to establish a reliable bond, especially in the dual-cure groups, but it could be seen that the self-cure groups performed worse in terms of shear bond strength.

Many authors have found that shear bond strengths to (bovine) dentin were lower after self-curing than after dual-curing of dual-cured composite materials [43-45]. Microtensile bond strength to dentin [3] and shear bond strength to composite [4] is also enhanced by dual-curing a dual-cured cement. A possible explanation for the phenomenon is a lower degree of conversion present in the specimens that had to rely on chemical initiators alone, as in the self-cure groups, resulting in lower mechanical properties of the cements. The weaker bond strength of the self-cure groups of RelyX Unicem in this study could also be explained by a difference in degree of cure of the specimens. The volumetric shrinkage of RelyX Unicem was significantly lower after self-curing compared to after dual-curing (Table 6.4). This is representative for a lower degree of cure. It has been shown that dual-curing RelyX Unicem results in a degree of conversion that is twice as high as compared to self-curing [22]. Higher degree of cure results in increased mechanical properties. This can explain the higher bond strengths and shear stresses of the dual-cured groups. The same effect can be observed for DC Core Automix, although neither the difference in volumetric shrinkage nor the difference in shear stresses are statistically significant at $\alpha = 0.05$. Apparently a relatively small increase of around 10% in volumetric shrinkage can reduce the bond strength of a cement by about 50%.

According to Pereira et al. [46] the curing reaction of Panavia F 2.0 also seems to depend on photo-polymerization, but in the presence of ED-primer, self-curing is possible because aromatic succinate salts from the primer diffuse into the polymerizing resin cement, which allows polymerization to occur [47, 48]. This is confirmed by the results of this study, since no differences between curing modes were observed in terms of bond strength and shear stresses for Panavia F 2.0. Light-curing through another restorative material is less effective than direct light-curing, but still more effective compared to self-curing in establishing a high degree of cure [49, 50]. The limited thickness of 1 mm of the composite discs would still have guaranteed an adequate light-curing of the cements.
The shrinkage stresses were much higher after dual-curing compared to self-curing, as demonstrated in Table 6.4. This has been reported in previous studies [21]. It can be explained by the faster polymerization process and reaching of the gel-point of these cements due to the light activation, after which there is less possibility for viscous flow of the cements to relieve stresses compared to the slow polymerization of self-cured cements.

Shrinkage stresses would also be expected to be higher in the DCC and DCD specimens compared to the DC specimens. Many articles have been written regarding the relationship between C-factor and bond strength. The general consensus is that a high C-factor has a negative influence on the bond strength [15, 51, 52], although not only the C-factor but also the characteristics of deep dentin as an adhesion substrate may contribute to lower bond strengths [53]. The use of a cement in a thin layer automatically results in a high C-factor cavity configuration with the disadvantages mentioned above. In thin resin layers in a low-compliance test set-up, the polymerization contraction stresses can achieve numbers of over 20 MPa [54]. On the basis of the C-factor alone, one might expect that the DCC and DCD groups would have lower bond strengths than their corresponding DC groups. However, it has also been shown that in a compliant set-up, in which the substrate materials are able to shrink towards each other, the contraction stress in thin resin films is highly reduced [18]. Even though the use of the cements in a thin layer (DCC and DCD groups) generally led to higher bond strengths compared to using the cements in bulk (DC-groups), this observation was not supported by FEA. Apparently the shrinkage stresses potentially caused by applying the cement in a thin layer were cancelled out by the compliance of the test set-up. This means that in this test set-up, the degree of cure of the cement had a higher influence on the bond strength and the maximum shear stresses compared to the contraction stresses.

The used test set-up could be a clinically relevant set-up for bonded onlays and crowns, where the restoration is able to move closer to the preparation as a result of the convergence of the preparation. In the case of a bonded post or inlay, however, the compliance of the tooth substrate or restoration is not likely to compensate for the volume loss of the cement as a result of volumetric shrinkage, which would result in high stresses and a reduced bond strength. Most in vitro studies use specimens with a C-factor of up to C = 5. Although this is useful for intracoronal restorations, it does not predict what would happen in the post space or underneath an indirect restoration,
where C gets a lot higher. In these cases, no compliance of the substrate is to be expected, and disruption of the cement layer from the dentin can indeed take place. As previously mentioned, when resin cements are applied in thin layers and in confined spaces, the contraction stress can exceed 20MPa [54], which makes disruption of the bond very likely. Braga et al. found that even in self-cure modes, the contraction stresses of dual-cure cements had enough magnitude to disrupt the bonding of the cement to dentin beneath Class I inlays [55]. In a confined situation, the microtensile bond strength of a resin composite to root dentin is lower compared to the microtensile bond strength in an open situation. Furthermore, premature failures of microtensile test specimens have been reported to be more common after setting of the cement in a confined space [16].

Conclusions

Shear bond strength failure loads used as input in FE models resulted in local stresses comparable to values reported for microtensile bond strength tests. Within the limitations of this study, it can be concluded that dual-curing leads to higher volumetric shrinkage for RelyX Unicem and higher contraction stress for all tested cements compared to self-curing 30 minutes after curing. Panavia F 2.0 proves to have lower volumetric shrinkage values compared to RelyX Unicem and DC Core Automix, and lower contraction stress values compared to RelyX Unicem and DC Core Automix in the dual-cure mode. Panavia F 2.0 shows lower shear stress values compared to the other two cements. Moreover, dual-curing instead of self-curing leads to higher shear stress values in the case of RelyX Unicem.
References


Shear bond strength of resin cements analyzed by FEA


