Evaluation of Marginal and internal adaptation of adhesive class II restorations, in vitro fatigue tests
Dietschi, D.L.

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Chapter 3:
Marginal and internal adaptation of class II restorations after immediate or delayed composite placement
Aim of the study

Base and liners supposedly act as stress absorbers or stress breakers during the insertion and polymerization of subsequent composite layers and afterwards, during functional loading. Among the numerous kinds of dental bonding agents, those which make use of a thick filled adhesive, applied separately from the primer, rely on to this "stress absorption" principle. In contrast, most of the very popular "one-bottle" adhesives allow only a very thin adhesive layer to be deposited on dentin. Although both types of adhesive systems provide bond strength values of similar range (May et al, 1997; Wakefield et al, 1998; Wilder et al, 1998; Tanumiharja et al, 2000), it remained to be determined if they provide the same restoration adaptation quality.

Apart from the influence of restorative materials and techniques, different parameters have to be considered to estimate the damaging potential of polymerization stresses (Carvahlo et al, 1996). Amongst the most important, are the configuration factor (Feilzer et al., 1987; Yoshikawa et al, 1999), the material properties (Kemp-Scholte and Davidson, 1990a), the cavity size, the presence or absence of enamel around cavity margins and the dentin quality, morphology and location (Perdigao et al, 1994; Shono et al, 1999). These parameters will determine how well adhesion to cavity walls, polymerization stresses and compensatory phenomena, such as flow and elastic deformations, balance each other. In clinical conditions, a satisfactory marginal and optimal internal adaptation might be difficult to achieve for large and deep restorations, due to an unfavourable combination of aforementioned elements.

The semi-direct and indirect techniques (Dietschi and Spreatifico, 1997) are aimed to solve this problem by confining composite polymerization shrinkage to the thin cementing gap, thus reducing the magnitude of stresses (Krejci et al., 1993; Dietschi and Herzfeld, 1998). Another advantage of this approach, although generally ignored, is the preservation of the adhesive interface during the intermediary or temporary phase and as well, an improved bond strength, all resulting from the delay preceding the cementation phase (Burrow et al, 1994; Burrow et al, 1996; Braga et al, 2000). This is likely to have a very positive influence on the restoration marginal and internal adaptation.

Therefore, the aim of the present study was to confirm the hypothesis that the delay between the adhesive application and composite built-up could have a significant impact on restoration adaptation. The hypothesis was tested for two different adhesive systems.

Specific materials and methods

Specimen preparation

Box-shaped Class II cavities (MOD) with parallel walls and bevelled enamel margins were prepared, with proximal margins located 1.0 mm below (mesially) and above (distally) the
cementum-enamel junction, such as described in chapter 2 (Fig. 2.5). The 40 prepared teeth were randomly assigned to one of the 4 experimental groups, corresponding to the 2 adhesive systems and the delay before composite placement.

**Restorative procedures**

The same restorative material (TPH Spectrum, DeTrey-Dentsply; Konstanz, Germany) but two different adhesive systems were evaluated (Optibond FL, Kerr; Orange, CA-USA; Prime & Bond 2.1, DeTrey-Dentsply).

After completion of the preparation, enamel was selectively etched for 30s prior to a 15s full cavity etching. The cavity was thoroughly rinsed for 30 s and gently air dried (3s air spray with low pressure) so that conditioned dentin was kept slightly moist. Then, adhesives were placed according to manufacturer’s instructions and light-cured for 40s.

The restorative material was either placed immediately following light-curing of the adhesive (**immediate placement: IP**) or after 24h (**delayed placement: DP**). The samples, of which filling was postponed, were stored in a damp container (sample in a water saturated atmosphere) to prevent sample dehydration. Group distribution is given in Table 3.1.

A horizontal layering method (Lutz and Kull, 1980) (Fig.3.1), was applied for the filling of proximal boxes (3 layers, the first one having a 1mm thickness) followed by the remaining occlusal volume. Each increment was separately cured for 40s with a halogen light-curing unit (Visilux XL 3000, 3M, St-Paul, MN-USA), the power density of which is about 525 mW/cm². The material characteristics and references are given in Table 3.II.

Flame and pear-shape fine diamonds burs (Intensiv No 4205L; 4255; 5205L and 5255) and polishing discs (Pop On XT, 3M; St-Paul, MN-USA) were used for restoration finishing and polishing.

Mechanical loading was applied such as described in chapter 2 (Figs. 2.1 - 2.4). The standardized preparation of specimen for SEM evaluation, detailed in chapter 2 ; figs. 2.6 - 2.10), was applied to all samples for the marginal and internal adaptation evaluation.

**Statistics**

All results were submitted to a parametric statistical analysis. The differences between the groups, before and after each fatigue phase, the evolution of the dentin marginal adaptation during mechanical loading and the differences between the groups and locations for the internal adaptation were explored by an ANOVA and Sheffe F test (as a multiple comparison test) (Sachs, 1974). All tests were carried out at a 95% level of significance.
Results

The results of the marginal adaptation evaluation (in percentage of continuity) are presented in Figs. 3.2 to 3.4 and in table 3.11, together with statistical analysis. The results of the internal adaptation evaluation (in percentage of continuity) are presented in Fig. 3.5 and table III; together with the statistical analysis.

Marginal adaptation

The adaptation to enamel proved satisfactory before, as well as after mechanical loading, on both restoration sides. Actually, percentages of enamel margins in continuity varied between 98.7% and 100% before loading and 87.2% and 97.6% after loading, with no significant difference among the experimental groups (table II). The only type of defect which was observed on enamel margins, however with low percentages, was the "marginal tooth fracture".

Following mechanical loading and as a result of marginal opening, the proportion of margins in continuity at the dentin level decreased in all groups [percentages of "continuity" ranging from 88.8% (OB-IP) to 100% (PB-IP) at baseline and from 55.1% (PB-IP) to 89% (OB-DP) after mechanical loading] with a significant change for PB-IP (between 0 and 500'000 or 1'000'000 cycles) and OB-DP (between 0 and 1'000'000 cycles). After 500'000 and 1'000'000 loading cycles, the use of Prime & Bond, together with an immediate composite insertion (PB-IP), resulted in a marginal adaptation on dentin significantly worse than following a delayed composite application, whatever adhesive was applied. Where adhesive failure were observed, the separation usually occurred between the restoration and the hybrid layer.

Internal adaptation

The restoration adaptation to enamel was excellent in all groups [ranging from 96.9% (OB-DP) to 100% (OB-IP)]. Regarding adaptation to dentin, the overall quality was also satisfactory [WDI percentages ranging from 79.2% (PB-DP) to 98.3% (OB-DP)] (Table 3.VI). Regarding the influence of the different locations on internal restoration adaptation, significant differences in "continuity" values were found only between the dentin occlusal area for PB samples, or the gingival dentin area for PB-DP, and gingival enamel. Optibond samples showed a better internal adaptation than Prime & Bond samples, at the gingival and occusal interfaces.

Micromorphological observations of internal interfaces

In most of samples, an acid resistant layer of 5 to 10 µm thickness was present between the restoration and the intact dentin, likely corresponding to the hybrid layer. Resin tags showed a variable penetration in the tubules, usually between a few to 50µ (Fig.3.6 and 3.7). The
protocol applied to the samples of this study did not allow to identify the bonding resin layer. No specific morphological feature could be attributed to any of the adhesives under evaluation; they both revealed similar variations in tag length and density or hybrid layer thickness.

Adhesive failures were found to occur at the top of the hybrid layer, in particular where no tag formation was visible (Figs. 3.8 and 3.9). The separation also predominantly occurred at the occlusal and gingival surfaces in the Prime & Bond samples. Other failure mechanisms such as cohesive fractures in dentin, within the hybrid layer, or a detachment of the hybrid layer at its base were virtually not found.

Discussion

Materials and method

The same restorative composite resin, a fine hybrid (TPH Spectrum), was used in all groups in order to reveal the influence on restoration quality of the adhesive type and the delay between adhesive placement and composite build-up.

Marginal adaptation to enamel

The restoration adaptation to mesial or distal enamel proved satisfactory in all groups, despite a severe mechanical fatigue test. Actually, very low proportions of defects were found at enamel margins, initially as well as after loading. Those observed proved to be mainly tooth micro-fractures. This very favourable finding likely reflects the influence of prism orientation in bonding efficiency to acid-etched enamel; it is known that a bevelled margin with enamel prisms cut roughly perpendicular to their long axis is a configuration more favourable than a butt margin (Munechika et al, 1984; Carvahlo et al, 2000). Actually, larger proportions of enamel micro-cracks were observed in in vitro mechanical loading tests conducted on cavities with a butt margin design (Krejci et al, 1993; Dietschi et al, 1998 and 1999). This speaks again in favour of placing a bevel around cavities to be filled with a direct technique, wherever enamel thickness is 1mm or more (Krejci, 1984; Dietschi et al, 1995a).

Adaptation to dentin

In dentin, significant differences in margin quality were observed, between placement methods and adhesives; the test hypothesis, namely that different types of adhesive or a delayed composite placement may influence restoration adaptation, was then confirmed. This difference in margin quality proved however significant only after 500'000 cycles. With consideration of the high initial percentages of continuous margins, this proves that both DBA were equally efficient in preserving initial interface integrity, but following simulated functional
stresses. This stresses the influence of mechanical loading in such in-vitro evaluations and the primary importance of fatigue tests in pre-clinical testing of adhesive techniques.

The samples of the PB-IP group, with immediate composite insertion and making use of the so-called "one-bottle" non-filled adhesive system (Prime & Bond 2.1), showed the smallest proportion of excellent margins. As well, the assessment of internal adaptation in dentin revealed more interfacial defects with this adhesive, compared to the filled system (Optibond FL). Different phenomenon, probably relating to each other, explain these findings. The first one is certainly the positive influence of the delay, before submitting the interface to polymerization or functional stresses. Actually, the bond strength is known to develop progressively, reaching its maximal value only after several hours (Burrow et al, 1994; Burrow, 1996; Braga, 2000). The co-polymerization process of the different monomers involved in the formation of the adhesive interface as a whole, necessitates time for completion. Immediate adhesion potential being then not optimal, one has to expect disappointing results in unfavourable configurations, such as deep and large cavities restored with a direct technique. As well, because most of data found in publications or manufacturer documents regarding maximal bond strength of dentin adhesives result from tests performed after 24h, clinicians tend to underestimate the occurrence of adhesive failures. Undoubtedly, more attention should be paid to this fact. For this reason, it can be advantageous to place a stress breaker layer in the system, which relates to the second explanation of the present results. Actually, the elastic deformation of a more "flexible" material (e-modulus lower than the restorative material) (Kemp-Scholte and Davidson, 1990b; Stavridakis et al, 2000) placed close to or within the adhesive interface might help to absorb immediate (resin polymerization shrinkage) and delayed strains (spontaneous post-polymerization and functional forces). The importance of the elastic" layer has been shown as early as 1990 (Kemp-Scholte and Davidson, 1990a and b); since then, several studies confirmed the validity of this concept (Davidson, 1994; Swift et al, 1996; Friedl et al., 1997; Choi et al, 2000; Dietschi et al, 2003).

A last potential explanation for the inferior efficiency of such a one-bottle adhesive is an insufficient or total lack of polymerisation of the very thin resin layer left after adhesive placement and solvent evaporation (only a few microns) (VanMeerbeeck, 1993). Actually, the inhibitory effect of oxygen is known to affect resin polymerisation to a depth of 100μm or more and to create a layer of totally non-polymerised resin of about 15μm (Rueggeberg and Margeson, 1990). As resin penetrates the demineralized dentin to a depth of only 0 to 10μm and considering the extremely thin resin coating normally persisting on the dentin surface (VanMerbeek et al, 1992; Prati et al, 1999), a complete resin polymerisation within the adhesive interface prior to composite placement is unlikely. As a consequence, the collagen network might be disturbed by composite placement, which generally results in a higher proportion of adhesive failures at the hybrid layer-bonding resin interface (Dietschi et al, 1995b; Tay et al,
Therefore, in a clinical class II configuration and in the absence of hybrid layer stabilisation, the resistance to stress of the adhesive interface promoted by a thin uncured resin layer is probably reduced. This sustains the findings of Frankenberger et al (1999) who showed that a pre-curing of the bonding resin was mandatory to maximise the quality of indirect and direct class II restorations.

In the present study, the restorations were realised following the total bonding concept (Krejci and Stravidakis, 2000), which requires adhesion to be established on all cavity surfaces. The rationale behind this concept is to provide an even stress distribution within the tooth-restoration system, assuming on the long-term behaviour alike natural teeth. Contrarily, with the concept of selective bonding, as described in 1986 by the group of Lutz and co-workers (1986a and b; Krejci and Stavridakis, 2000) adhesion is established only at the margins, providing a large internal free surface. In a critical class II configuration, when polymerisation stresses might develop faster and higher than adhesion, excessive internal stress build-up is prevented by separation at a pre-determined interface. The separation takes place between a base-lining made of glass-ionomer and the next composite layer, while following the original technique (Lutz et al, 1986b), or between two different kinds of dentinal adhesives (Krejci and Stavridakis, 2000). The selective bonding efficacy is based on the preservation of dentin biological seal by either glass ionomer or dentin adhesive and, as well, by the lack of direct stress on the tooth-lining interface. The concept of delaying stress development follows the same objectives, when the total bonding concept is applied to a critical cavity configuration. In this situation, the indirect technique is to be favoured, which allows postponing and reducing stresses developing at the tooth-restoration interface. As a concentration of functional stresses along the margins, as resulting from the strict application of the selective bonding concept, might be detrimental to restoration quality (Thonemann et al, 1999), the use of indirect or semidirect techniques (Dietschi and Spreafico, 1997) for large and deep class II cavities still appears suitable.

**Micromorphology of the internal adhesive interface**

The micro-morphology of the internal adhesive interface, as shown on the gold-sputtered resin replicas, is compatible with the description made by Nakabayashi (1982), Pashley (1992) and Van Meerbeek (1993). SEM observation of sample sections at higher magnification revealed that adhesive failures chiefly occurred above the hybrid layer and predominantly where resin tag formation was insufficient. This sustains the importance of tags in improving bond to dentin (Pashley, 1992).

In other areas of debonding, the influence of the substrate cannot be totally excluded. However, the use of non-caries intact teeth, as well as the absence of clear differences in gap percentages between the different cavity zones, does not make it a likely variable. Although cohesive fractures in dentin were observed after bond strength tests, especially micro-tensile
bond strength tests (Amstrong et al, 1998; Schreiner et al, 1998; Yoshikawa et al, 1999), this failure mode was not pertinent to the present study. This confirms the idea that actual bond strength of modern dentin adhesive do not surpass the yield strength of resin composites or the ultimate tensile strength of dentin (Tay et al, 2000), especially in a clinically relevant configuration.

**Conclusions - clinical relevance**

In the present experimental conditions, it can be concluded that:
- adaptation to enamel was satisfactory with bevelled proximal margins
- the marginal adaptation to dentin was better after a delayed placement of the restorative material, showing that the resistance of the adhesive interface to polymerisation and functional stresses is not optimal immediately after DBA application
- the internal adaptation to dentin proved better when creating a thick adhesive layer (Optibond FL), confirming the importance of a stress-releasing layer within the adhesive interface
- debonding took place predominantly over the hybrid layer; this interface was again identified as the weak link of dentin adhesion.
Table 3.1: Composition of Products under investigation (manufacturer’s data)

<table>
<thead>
<tr>
<th>Products</th>
<th>Product name (manufacturer)</th>
<th>Composition</th>
<th>Batch numbers</th>
</tr>
</thead>
<tbody>
<tr>
<td>conditioner</td>
<td>Ultraetch (Ultradent; Salt-lake City, USA)</td>
<td>H$_3$PO$_4$ 37% gel</td>
<td>-</td>
</tr>
<tr>
<td>adhesive I</td>
<td>Optibond FL (Kerr, Romulus, CA-U.S.A.)</td>
<td>Primer: 2 (hydroxyethyl) methacrylate (HEMA)</td>
<td>712501</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Glycerol phosphate dimethacrylate (GPDM)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Mono (2-methacryloxy ethyl) phtalate</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Ethyl alcohol</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Water</td>
<td></td>
</tr>
<tr>
<td>adhesive II</td>
<td>Prime &amp; Bond 2.1 (Dentsply De Trey; Kontsanz, Germany)</td>
<td>Dymethacrylate resins</td>
<td>711352</td>
</tr>
<tr>
<td></td>
<td></td>
<td>PENTA (dipentaerythritol penta acrylate monophosphate)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>photoinitiators, stabilizers</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>cetylamine hydrofluoride</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>acetone</td>
<td></td>
</tr>
<tr>
<td>restorative material</td>
<td>TPH spectrum (Dentsply De Trey; Kontsanz, Germany)</td>
<td>mod. BisGMA, BisEMA, TEGDMA</td>
<td>961016</td>
</tr>
<tr>
<td></td>
<td></td>
<td>barium alumino boro silicate glass</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>colloidal silica</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Initiators</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>stabilizers</td>
<td></td>
</tr>
</tbody>
</table>
Table 3.11: Results of the marginal adaptation evaluation at the different proximal locations, according to the number of mechanical loading cycles (percentages of continuity +/- SD)

<table>
<thead>
<tr>
<th>Nb of cycles</th>
<th>location</th>
<th>PB-IP</th>
<th>PB-DP</th>
<th>OB-IP</th>
<th>OB-DP</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Enamel (distal)</td>
<td>98.9 (3.2)</td>
<td>100 (-)</td>
<td>99.5 (1.4)</td>
<td>98.7 (2.5)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>98.9 (3.2)</td>
<td>99.4 (1.8)</td>
<td>99.5 (1.4)</td>
<td>98 (2.8)</td>
</tr>
<tr>
<td>250'000</td>
<td></td>
<td>96.6 (5.7)</td>
<td>98.6 (2.9)</td>
<td>98.5 (2.3)</td>
<td>95.7 (5.4)</td>
</tr>
<tr>
<td>500'000</td>
<td></td>
<td>94.5 (7.1)</td>
<td>87.2 (31.3)</td>
<td>94.9 (5.5)</td>
<td>93.0 (7.4)</td>
</tr>
<tr>
<td>1'000'000</td>
<td></td>
<td>100 (-)</td>
<td>100 (-)</td>
<td>98.8 (3.5)</td>
<td>99.6 (0.7)</td>
</tr>
<tr>
<td>0</td>
<td>Enamel (mesial)</td>
<td>100 (-)</td>
<td>99.4 (1.2)</td>
<td>100 (-)</td>
<td>99.2 (0.7)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>100 (-)</td>
<td>99.0 (1.8)</td>
<td>99.1 (1.6)</td>
<td>98.8 (1.7)</td>
</tr>
<tr>
<td>250'000</td>
<td></td>
<td>95.2 (13.4)</td>
<td>97.6 (4)</td>
<td>93.6 (13.8)</td>
<td>95.0 (10.3)</td>
</tr>
<tr>
<td>500'000</td>
<td></td>
<td>100 A (8.4)</td>
<td>96.5 (31.8)</td>
<td>88.8 (31.8)</td>
<td>99.8 A (0.7)</td>
</tr>
<tr>
<td>1'000'000</td>
<td></td>
<td>78.1 A,B (10)</td>
<td>95.5 (10.4)</td>
<td>91.4 (16.7)</td>
<td>97.0 B,A (5.8)</td>
</tr>
<tr>
<td>0</td>
<td>Dentin</td>
<td>69.5 B,a (14.4)</td>
<td>91 b (11.6)</td>
<td>85.1 a,b (16.5)</td>
<td>95.4 B,A,b (5.3)</td>
</tr>
<tr>
<td>250'000</td>
<td></td>
<td>55.1 B,a (22.6)</td>
<td>86.9 b (10.1)</td>
<td>78.0 a,b (19.1)</td>
<td>89.0 B,b (9.1)</td>
</tr>
</tbody>
</table>

For comparison between groups (rows), means with same lower case letter are not statistically different at p=0.05 using the Scheffé F-test. For comparison between the number of cycles (columns), means with same capital letter are not statistically different at p=0.05 using the Scheffé F-test. No significant difference was found for rows and columns without letter.
Table 3.III: Results of the internal adaptation evaluation, according to the different interface segments and the whole dentin interface (total) (percentages of continuity +/- SD)

<table>
<thead>
<tr>
<th>Groups</th>
<th>GE</th>
<th>GD</th>
<th>AD</th>
<th>OD</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>PB-IP</td>
<td>98.8 a</td>
<td>83.4 A,B,a,b</td>
<td>98.0 a,b</td>
<td>68.4 A,b</td>
<td>86.3 (13.3)</td>
</tr>
<tr>
<td></td>
<td>(3.5)</td>
<td>(12.8)</td>
<td>(12.6)</td>
<td>(34)</td>
<td></td>
</tr>
<tr>
<td>PB-DP</td>
<td>98.6 a</td>
<td>67.1 A,b</td>
<td>91.5 a,b</td>
<td>68.7 A,b</td>
<td>79.2 (17.4)</td>
</tr>
<tr>
<td></td>
<td>(4)</td>
<td>(27.1)</td>
<td>(10.9)</td>
<td>(24.5)</td>
<td></td>
</tr>
<tr>
<td>OB-IP</td>
<td>100</td>
<td>93.7 B</td>
<td>98.3</td>
<td>95.9 A,B</td>
<td>97.4 (3.8)</td>
</tr>
<tr>
<td></td>
<td>(-)</td>
<td>(13.6)</td>
<td>(3.8)</td>
<td>(6.8)</td>
<td></td>
</tr>
<tr>
<td>OB-DP</td>
<td>96.9</td>
<td>98.7 B</td>
<td>99.8</td>
<td>97.4 B</td>
<td>98.3 (2.6)</td>
</tr>
<tr>
<td></td>
<td>(5.9)</td>
<td>(3)</td>
<td>(0.5)</td>
<td>(3.4)</td>
<td></td>
</tr>
</tbody>
</table>

For comparison between products (columns), means with same lower case letter are not statistically different at p=0.05 using the Scheffé F-test. For comparison between locations (rows), means with same capital letter are not statistically different at p=0.05 using the Scheffé F-test. No significant difference was found for rows and columns without letter.
Figure 3.1: Horizontal layering technique (1-3 represent the 3 subsequent increments).
Figure 3.2: Results of the marginal adaptation evaluation, in enamel, at the sample mesial surface, before and after the different loading phases.

Figure 3.3: Results of the marginal adaptation evaluation, in enamel, at the sample distal surface, before and after the different loading phases.
Figure 3.4: Results of the marginal adaptation evaluation, in dentin, at the sample mesial surface, before and after the different loading phases.

Figure 3.5: Results of the internal adaptation evaluation, after mechanical loading.
Figure 3.6: Optibond FL sample - immediate composite placement. The adaptation is in "continuity" with a well organized hybrid layer and resin tags clearly visible.

C = composite restoration;  D = dentine;  HL = hybrid layer;  RT = resin tags

Figure 3.7: Prime & Bond sample - delayed composite placement. The micromorphology of the adhesive interface is very similar to the one of Optibond FL, showing a well organized hybrid layer and resin tags.
Figure 3.8: Prime & Bond sample - immediate composite placement. The gap clearly occurred over the hybrid layer, in an area with no resin tag formation. This points out the importance of tags in bonding efficiency.

C = composite restoration; D = dentine; HL = hybrid layer; G = gap

Figure 3.9: Optibond FL sample - immediate composite placement. Similar observation were made underneath gap in this groups.
References


SCHREINER RF, CHAPEL RP, GLAROS AG, EICK JD. Microtensile testing of dentin adhesives. Dent Mat 1998; 14; 192-201.


