Dentin bonding agents. Relevance of in vitro investigations

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Abstract: Composite resin restorations placed into intradental cavities often show incomplete marginal adaptation due to the effects of polymerization contraction stresses and due to mechanical and/or thermal stress generated at the restoration/cavity interface. Bonding of restorative resins by micromechanical retention to acid-etched enamel and by chemical coupling with dentin and root cementum has a potential of reducing or eliminating marginal gap formation. Dentin bonding agents are usually evaluated by in vitro testing procedures. The determination of bond strength to dentin is the most commonly used method. Bond strength figures should be considered relative to the influence of experimental variables and relative to their reliability as parameters for prediction of cavity adaptation. The efficacy of dentin bonding agents is not conclusively described by in vitro testing. Bond strength figures are only roughly discriminating parameters and not very predictive for the long term clinical performance of a bonded resin restoration.

Key words: Bond strength; dentin bonding agents; restorative dentistry.

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INTRODUCTION

The restoration with composite resin of lesions which are not totally surrounded by enamel is a particular challenge to restorative dentistry. Defects of this type are often CI II, sometimes extensive CI III cavities and almost regularly cervical tooth lesions. The treatment needs for such lesions, either cervical erosions or root caries, is steadily increasing as documented by Katz et al.1

Until recently, restoration of this type of defect with restorative resins and cements has relied on a cavity design giving mechanical retention by undercuts. Consequently, a considerable amount of sound tooth substance had to be sacrificed resulting in a weakening of the tooth. A firm adaptation, however, between restoration and cavity wall was not obtained.

The development of glass ionomer cements has led to some improvements in the restoration of these lesions. The advantages of glass ionomer cements are that they form adhesive bonds to calcified tissues and release fluoride which is readily taken up by mineralized dental tissues. The disadvantages are their brittleness, opacity, rather large water sorption and surface roughness.

The introduction of dentin bonding agents, which by a chemical coupling mechanism mediate a bond between the dental hard tissue; either to the inorganic or to the organic part of dentin, and the restorative resin, has been announced as a real breakthrough in dentistry. After several years of experience with some of these compounds, it became obvious that the high initial expectations, which were mainly based on the results of laboratory studies, were not being fulfilled. New dentinal bonding agents have become available to the profession recently. The manufacturers compete for a share of their products in the market; however, as before, the claims for efficacy are almost exclusively based on the results of laboratory investigations. Sometimes it seems that the manufacturers are competing in a world championship in bond strength figures, where it is implied that the effectiveness of a product is coupled with the magnitude of the bond strength. Similarly, microleakage studies in extracted teeth are often cited as the ultimate proof of a product's benefit.

The purpose of this paper is to discuss the clinical significance of in vitro evaluation parameters, particularly of bond strength figures.

Bond strength to dentin has been and is still the most commonly reported parameter for the evaluation of dentin bonding systems. There is a wide variation in the preparation and design of test specimens. However, basically a flat dentin surface is prepared by grinding and a composite resin rod is polymerized on the dentin following pretreatment and bonding agent application. Then shear or tensile bond strengths are determined. The bonding interface is only moderately loaded by polymerization contraction stresses parallel to the flat dentin surface while shrinkage in a perpendicular direction can occur almost freely and without generation of stresses at the bonding site. This stress pattern is very different from the one developed in a cavity where con-
considerable stresses are generated which often result in debonding.

The wide range of bond strength figures reported for identical material systems indicate that the test is very complex. Possible variables introduced during bond strength testing are as follow:

Age of the extracted teeth

The age of the extracted teeth, i.e. the duration of storage prior to specimen preparation seems to be of minor significance to bond strength. No differences were found in our investigations when extracted teeth were used either immediately after extraction or following storage of up to three months.

Storage condition of the extracted teeth

In contrast, the storage condition of the teeth may be a relevant variable. It is, for example, known that preservation in a thymol solution can have an adverse effect on the degree of polymerization of resins. Hansen & Asmussen demonstrated that dentin which had been exposed to eugenol-containing temporary restorations contained free eugenol even after refinishing the surface. Eugenol inhibits the polymerization of resins and will thus have an adverse effect on the bond strength to dentin.

In a recent publication, very low bond strength figures have been reported for the Gluma bonding system. In this study the extracted teeth had been stored first in 10% NaOCl solution for 24 hours and then in a 5% NaOCl solution for non-specified time periods. NaOCl effectively removes connective tissue and collagen to which Gluma is suggested to bond chemically. Therefore, it is not surprising that low bond strengths were reported.

Physiologic saline is suitable when teeth are stored for a few days only, while a 1% Chloramine solution according to Jorgensen et al is an adequate preservative storage medium.

Human or bovine teeth

Due to difficulties experienced in obtaining extracted human teeth, bovine dentin is often used as an alternative. The strength figures obtained on bovine teeth, however, are not comparable and are commonly lower than the ones recorded on human dentin.

Site of dentin used for bonding

Another major variable in bond strength tests is the site of dentin as reported by Causton and Mitchem & Gronas. Suzuki & Finger found a linear relationship between the remaining dentin thickness to the pulp and the shear bond strength mediated by the Gluma/Lumifor system, by Clearfil New Bond/Clearfil Ray and by Scotchbond 1.c./Silux when determined 15 minutes after light activation of the restorative resin. The relationship between the remaining dentin thickness and the percentage area of solid dentin, that is the area of dentin minus the percentage area of dentinal tubules, was described by a logarithmic regression line. The bond strength on dentin close to the pulp was only 30-40% of the bond strength recorded on peripheral dentin. These results may to some extent explain the high coefficients of variation often associated with mean bond strength figures. For clinical dentistry, however, the findings may have very limited, if any, significance. When the rules of adhesive cavity preparation are followed, the area of exposed dentinal tubules constitutes a small part only of the dentin surface covered by the restorative. This holds for both smooth surface cervical erosion lesions and for dentin underneath an area of dental caries. In these instances, the lumina of the dentinal tubules are very narrow or may even be obliterated by the apposition of irregular dentin.

In an additional investigation, it was demonstrated that the bond strengths obtained on non-carious extracted third molars were considerably less than the strengths recorded on a random sample of extracted teeth. The reason for this finding is not fully understood. However, it is hypothesized that non-carious third molars are usually extracted from young individuals soon after eruption. These teeth may have wider dentinal tubules and thus a decrease in the effective bonding area and may be less mineralized than teeth which have been exposed to the oral environment for longer time periods.

Roughness of the dentin surface

Mowery et al studied the effect of the surface roughness of dentin samples on the shear bond strength of light cure Scotchbond combined with Prisma Fil. They reported that a rough dentin surface prepared on 60 grit abrasive yielded 4.7 MPa in bond strength after 24 hours. On less roughened dentin abraded 260, 600 and 1200 grit abrasives significantly lower strengths were found. The lowest bond strength figure (2.7 MPa) was reported for dentin prepared on grit 600 abrasive. These observations may be of clinical significance in selecting the cutting instruments for cavity preparation.

Manabe & Finger evaluated the effect of dentin roughness on bond strength for the following systems: Clearfil n.b./Clearfil-Ray CL, Gluma/Lumifor (GL), Scotchbond 2/Valux (SV), Scotchbond/Silux (SS), and Tenure/Silux (TS). The tensile bond strengths were determined five minutes after the initiation of light activation of the restorative resins on dentin surfaces ground in one direction on silicone carbide paper with grits of 180, 240, 320, 400, 600 and 4000, respectively. With each of the five systems tested, the bond strengths were independent of the surface roughness of the underlying dentin. Testing by ANOVA showed no statistically significant differences (P > 0.05). The effective bonding surfaces produced by grinding of the dentin on each of the six grits were determined from the surface roughness profiles. The maximum effective dentin surface enlargement due to roughness was approximately 10%. This moderate increase in area may explain why...
no significant differences in bond strength were found in spite of the tremendous differences in surface roughness recorded in terms of R₅ or R₁₀ figures. The average pooled tensile bond strength figures in MPa from 60 determinations each were: CL 9.1, GL 15.5, SV 12.9, SS 4.2, and TS 10.1. From this investigation it was concluded that the roughness of prepared dentin surfaces might not be a variable of clinical significance for bonding of resins.

**Time of application of the load on the bonded specimen**

The time of the application of the load on the bonded test specimen is a critical factor. Bond strengths are usually determined after 24 hours' storage in water or saline at body or room temperature. The crucial time for measuring bond strength is, however, the early time when the stresses arising from polymerization contraction are established, since this is the time interval when debonding may occur between cavity wall and restorative material. It is suggested, therefore, that bond strengths should be determined immediately after the initial curing. Additionally, it is certainly necessary for a long term evaluation of the bonding efficacy to determine the bond strengths after prolonged storage time intervals to investigate whether the bond strengths are subject to changes, for example, by hydrolysis. Finger & Ohsawa reported that the bond strength of Clearfil New Bond/Clearfil-Ray 2 to 3 minutes after light activation of the composite resin was more than twice the bond strength recorded after 24 hours' storage in water.

Fasbinder et al reported that the bond strengths to dentin of Universal Bond/Prismafil, Bondlite/Command, and Scotchbond/Silux were significantly reduced by storage in water for six months. In contrast, Tenure/Ultrabond was not affected. The tensile bond bond strengths of this system after 24 hours and 6 months were 2.46 and 2.97 MPa, respectively. Munksgaard & Asmussen reported that the tensile bond strength of Gluma® bonded specimens stored in water was consistently maintained at 17.5 MPa throughout an observation period of six months.

**Thermocycling prior to testing**

The recent ADA guidelines for dentinal bonding agents propose that bond strength should be determined both after storage in water and after thermocycling of the specimens. Chan et al, O'Brien et al, and Marchman et al reported that thermocycling of the bonded test specimens had no adverse effect on bond strength. When tested according to the guidelines, there was a difference in bond strength for Gluma/Lumifor® specimens with or without thermocycling (Table). However, from a practical point of view these differences are insignificant.

**Tensile or shear loading**

Bond strength testing is usually done by the application of tensile or shear loading. Depending on the mode of loading and the individual experimental procedure, the test results may differ widely. The stress patterns generated are very complex and variable as demonstrated by von Noort et al. Bond strength figures depend very much on the individual method used. It is only reasonable to directly compare the bond strengths of different bonding systems when the methods used are identical. Tensile and shear loading may give very different results.

**Degree of curing of the bonding system**

The degree of curing, that is the degree of conversion of the double bonds in the resin polymer, has a strong impact on bond strength. Munksgaard et al reported that experimental resins containing propanal enhanced the shear bond strength when used in combination with the Gluma® dentin bonding agent. It is suggested that the propanal as a reducing agent may reduce oxygen inhibition of the polymerization and thereby increase bonding by the adhesive. Hansen demonstrated that light activation of Scotchbond® for 50 seconds instead of 10 seconds gave significantly reduced marginal gap widths in cylindrical butt-joint cavities. This, as well as the findings of Hansen & Asmussen that postponing polishing of the marginal area of a dentin bonded composite resin restoration improves the marginal quality, underline the clinical relevance of the degree of curing.

**Table 1. Tensile bond strength (MPa) of Gluma/Lumifor on dentin (X̄ + δ, n=60)**

<table>
<thead>
<tr>
<th></th>
<th>15 minutes</th>
<th>24 hours</th>
<th>30 days</th>
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<tbody>
<tr>
<td>Storage in H₂O</td>
<td>19.2 ± 3.5</td>
<td>23.1 ± 2.5</td>
<td>29.3 ± 3.8</td>
</tr>
<tr>
<td>(8/2/0)</td>
<td>(5/4/1)</td>
<td>(0/7/3)</td>
<td></td>
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<tr>
<td>Storage in H₂O</td>
<td>16.9 ± 3.5</td>
<td>21.2 ± 3.6</td>
<td>26.3 ± 4.2</td>
</tr>
<tr>
<td>plus thermocycling**</td>
<td>(9/0/1)</td>
<td>(5/3/2)</td>
<td>(0/8/2)</td>
</tr>
</tbody>
</table>

* The numbers in parentheses give the number of specimens out of ten which showed: mixed failure/cohesive failure in resin/cohesive failure in dentin.
** Thermocycling in H₂O between 55°C and 55°C; dwell times, 15 seconds; n=250.

Bond strength figures are commonly reported without referring to the mode of failure within the fractured specimens. The term "bond strength" implies that debonding occurred as a result of adhesive failure at the interface. For the more effective bonding agents, however, the fracture is often located within the restorative resin or within dentin, indicating that the true interfacial bond strength is higher than the figure reported.

Often microscopic inspection of the fracture morphology on the dentin side does not clearly reveal the nature of the failure. A proven method for assessment of the type of failure is 1) to etch by phosphoric acid and then 2) to clean, with a NaOCl-solution, the dentin side of the fractured bonding specimen, following sectioning of the tooth perpendicular...
no significant differences in bond strength were found in spite of the tremendous differences in surface roughness recorded in terms of $R_s$ or $R_p$ figures. The average pooled tensile bond strength figures in MPa from 60 determinations each were: CL 9.1, GL 15.5, SV 12.9, SS 4.2, and TS 10.1. From this investigation it was concluded that the roughness of prepared dentin surfaces might not be a variable of clinical significance for bonding of resins.

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|                | 15 minutes | 24 hours | 30 days |
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| Storage in $H_2O$ plus thermocycling $^{**}$ | $16.9 \pm 3.5$ | $21.2 \pm 3.6$ | $26.3 \pm 4.2$ |

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lar to and through the center of the debonded area. Fig. 1 shows an adhesive failure resulting from debonding of a Scotchbond 1.c./Silux specimen. There is almost no resin left on the bonding surface. The corresponding SEM picture for Clearfil New Bond/Clearfil-Ray® (Fig. 2) shows a mixed type of failure partially cohesive, but mainly adhesive debonding. In contrast, Figs. 3, 4 and 5, the SEM photographs for Gluma/Dentin Bond/Lumifor, Scotchbond 2/Valux, and Tenure/Silux respectively, show consistently cohesive failure in the resin. The SEM pictures demonstrate that bond strength figures have to be considered very critically, since generally they do not properly describe the quality of the bond. Thus, they may sometimes only reflect strength characteristics of the restorative resin system. It is also well-known that combination of one dentin bonding agent with different restorative resin systems may give different bond strength figures.11,24

It has been questioned whether in vitro bond strength testing will reflect the bond strength to dentin in vivo. Due to the hydrostatic pressure of the dentinal fluid in the tubules, a freshly cut dentin surface will be wet. This wetness may impair bonding. Pashley et al22 reported that no significant differences were observed between bonds made in vivo on dog teeth and those made in vitro on the same teeth 30 minutes, one day, one week and one month post-extraction. In spite of the similarity of the in vivo and in vitro bond strength results, the clinical significance of bond strength figures remains questionable.

Komatsu & Finger11 reported a logarithmic relationship between the maximum widths of marginal contraction gaps in cylindrical butt-joint dentin cavities (φ 3.0 mm, h = 1.5mm) and the early shear bond strength of a series of material combinations (r = -0.88).
The cavities were prepared with a 90° cavosurface angle and restored with different restorative resin systems in combination with different dentin bonding agents.

The 95% interval of prediction around the regression line was very wide, thus proving that strength figures do not adequately delineate the effectiveness of dentin bonding agents when used in cylindrical dentin cavities. Estimation of the early shear bond strength necessary to prevent gap formation in the above mentioned type of cavity gives a figure of 20 MPa, approximately. Similar estimates were reported by Munksgaard et al. and by Asmussen & Munksgaard. It is, however, important to recall that these correlations only hold under the specific conditions of the individual experimental procedures described in the respective studies. They are not valid in a more universal sense.

CONCLUSIONS

The following conclusions may be drawn:

1. Bond strength figures of different sources are not readily comparable.
2. Bond strength figures are only roughly discriminating parameters for the evaluation of a dentin bonding agent's efficacy.
3. Bond strength figures are only poorly correlated with in vitro cavity tests.
4. Bond strength figures should be reported together with the mode of failure of the specimens.
5. Bond strength testing procedures should be standardized.

Unfortunately, there is no documented relationship between the results of currently practiced in vitro evaluation methods for dentin bonding agents and their long term in vivo performance. Products which exhibit a low bond strength in the laboratory are unlikely to offer sufficient retention of a bonded restoration in vivo. In contrast, products which show a high bond strength immediately after curing of the restorative, and where debonding preferentially occurs as a result of cohesive failure in the restorative or in the dentin, are more likely to be clinically acceptable in the long run.

Although bond strength is proven not to be a reliable parameter for prediction of clinical performance of a dentin bonding agent, it makes sense to select a product for clinical use on the basis of its bond strength, as long as no more relevant long term evaluation data are available to the profession.

REFERENCES