Evaluation of the bond strength of different bracket-bonding systems to bovine enamel

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SUMMARY In an experimental study the bond strength of stainless steel, ceramic and plastic brackets to bovine enamel was investigated by tensile testing. The brackets were cemented to the enamel using a conventional two-paste orthodontic bonding resin, a light-cured, fluoride-releasing adhesive, a no-mix-orthodontic bonding resin and a light-curing glass ionomer cement.

For evaluation of the experimental data the Weibull analysis was applied. The highest values for the Weibull modulus \( m \) and the 10 per cent probability of failure \( \sigma_{10} \) were found in the tested plastic brackets (Dentaurum Edgewise plastic bracket and Spirit bracket) using a no-mix orthodontic bonding resin (System 1). However, the tensile stresses for the 90 per cent probability of failure \( \sigma_{90} \) were over 10 MPa and carried the danger of enamel fracture. Bracket bonding with glass ionomer cement cannot be recommended because of the low bond strength values for the 10 per cent probability of failure \( \sigma_{10} \). The most favourable bracket-bonding system concerning the Weibull modulus \( m \), the 10 and 90 per cent probabilities of failure \( \sigma_{10} \) and \( \sigma_{90} \) and aesthetics was the ceramic bracket with the silane-treated base (Allure III) using the light-cured, fluoride-releasing orthodontic bonding resin (Sequence).

Bond fracture occurred predominantly between bracket and orthodontic bonding resin, with two exceptions. Concerning the ceramic bracket with the silane-treated base (Allure III) using the light-cured glass ionomer cement (Photac Fil), there was no preferential site of failure. Regarding the ceramic bracket with the silane-treated base (Allure III) using the light-cured, fluoride-releasing orthodontic bonding resin (Sequence), bracket fracture was seen more often than bond separation between bracket and enamel. When the bond failure was located at the enamel–orthodontic bonding resin interface enamel prisms could be identified on the adhesive site by scanning electron microscopy.

Introduction

Since its introduction direct bonding of orthodontic brackets has become more and more important because of on-going development of the bonding technique, aesthetics and design parameters of the bracket bases (Swartz, 1988; Winchester, 1991; Droese and Diedrich, 1992; Fischer-Brandies et al., 1992; Bauer et al., 1993; Eberhard et al., 1994). Fundamental research including scanning electron microscope examination has been undertaken on direct bonding (Diedrich, 1979, 1981a, 1983; Dickinson and Powers, 1980; Diedrich et al., 1986; Fischer-Brandies et al., 1989; Britton et al., 1990; Winchester, 1991).

A high bond strength of the orthodontic bracket to enamel and a low failure rate are the basic demands for a bracket-bonding system (Diedrich, 1981b). Therefore, special requirements have to be fulfilled by the orthodontic adhesive and the design of the bracket base (Weißenberg and Diedrich, 1987; Ostertag et al., 1991).
In the present experimental study the bond strength of nine different bracket-bonding systems to bovine enamel was determined by tensile testing. The fracture stress values were evaluated by the Weibull analysis.

The Weibull analysis

Orthodontic bonding resins are brittle materials and produce a wide scattering of the bond strength data (Mojon et al., 1989). The Weibull analysis helps to handle these characteristics of the orthodontic adhesives. It enables the researcher to come to a more realistic evaluation of the bond strength than can be achieved by using normal distributions (Weibull, 1951; Ashby and Jones, 1986). The fracture stress values are not normally distributed and are better subjected to a Weibull analysis (Britton et al., 1990).

The Weibull equation depends on two parameters:

(i) The Weibull modulus. This can be compared to the standard deviation of a normal distribution. A low Weibull modulus indicates a wide scatter in the experimental data. A high Weibull modulus indicates a close grouping of the fracture stress values and a high level of reliability of the samples.

(ii) The characteristic level. This refers to the tensile stress at which 63.2 per cent of the samples fail. The characteristic level is similar to the mean value of the normal distribution. The higher the characteristic level the higher the bond strength of a bracket-bonding system (Weibull, 1951; McCabe and Walls, 1986; Britton et al., 1990).

The Weibull analysis enables the researcher to check if a bracket-bonding system is within required limits of bond strength. It is possible to predict the probability of failure of a sample at any level of tensile stress, e.g. tensile forces that are likely to be applied in orthodontic treatment (McCabe and Carrick, 1986; McCabe and Walls, 1986). The Weibull analysis shows that there is a certain probability of bond failure at low tensile forces even for a bracket-bonding system of high mean tensile strength. Therefore, it may sometimes be preferable to choose a bracket-bonding system with a slightly lower mean tensile strength but a higher Weibull modulus. The latter indicates a closer grouping of the data and a shorter tail of bond fractures at low stress levels (Ashby and Jones, 1986; McCabe and Carrick, 1986; Nkenke et al., 1993).

The Weibull analysis helps to identify combinations of brackets and adhesives that tend to fail at low stress levels. Clinical trials have previously only been carried out to determine the failure rates of bracket-bonding systems. These failure rates vary from 2 to 25 per cent or more (Diedrich, 1981b; Mizrahi, 1983; Weißenberg and Diedrich, 1987).

Due to the wide scatter of the bond strength data, it is necessary to test at least 20–30 specimens to predict the performance of a bracket-bonding system accurately with the Weibull analysis (McCabe and Walls, 1986).

Materials and methods

Five different types of brackets were bonded to 450 freshly extracted bovine teeth which had been polished with pumice (Table 1). Three orthodontic bonding resins and one glass ionomer cement were used (Table 2). To obtain a control bracket-bonding system, a stainless steel bracket with a mesh base (Diamond) was cemented to the bovine enamel using a conventional two-paste orthodontic bonding resin (Concise).

The teeth were embedded in acrylic resin and the finished samples were stored in a saline solution for 72 hours at room temperature. Tensile testing was then carried out using a universal testing machine (Universalszugprüfmaschine 1425; Zwick, Ulm, Germany) at a crosshead speed of 0.2 mm/min (Jähnig and Henkel, 1990) (Figure 1). The tensile strength was measured in Newtons and transformed into MPa by dividing the load by the area of the bracket base.

The bond strength data were subjected to a Weibull analysis. The Weibull equation (equation 1) allows the prediction of the probability of failure of a bracket-bonding system at any level of tensile stress (Weibull, 1951; McCabe and Carrick, 1986; McCabe and Walls, 1986).
where $P_f = \text{the probability of failure}$, $\sigma = \text{the tensile strength (Mpa)}$, $\sigma_u = \text{the threshold stress (MPa)}$ (tensile stress at which the first bond failure occurs). When sufficiently large numbers of samples have been tested, $\sigma_u$ approaches 0. Therefore, for most applications it is assumed that $\sigma_u = 0$, $\sigma_0 = \text{the characteristic level (MPa)}$ (tensile stress at which 63.2 per cent of the samples fail) and $m = \text{the Weibull modulus}$.

Taking natural logarithms twice, equation (1) gives a straight line with a slope that is the Weibull modulus ($m$) (Figure 2a and b). From these diagrams the characteristic level $\sigma_0$ (i.e. 63.2 per cent probability of failure) can be determined. In Figure 3 the probability of failure ($P_f$) is plotted against the tensile strength. The parameters and the application of the Weibull analysis have already been described in detail by Nkenke et al. (1993).

After tensile testing the fractured samples were examined with a scanning electron microscope.

**Results**

The parameters of the Weibull analysis calculated from the experimental data are summarized in Table 3 and Figures 2 and 3. The stainless steel bracket with the mesh base (Diamond) cemented to the enamel using the conventional two-paste orthodontic bonding resin (Concise) achieved the highest mean tensile strength ($\sigma_m = 8.04 \text{ MPa}$). The Weibull modulus
had an intermediate value amongst the brackets tested. There was a 10 per cent probability of failure ($\sigma_{10}$) at 4.00 MPa and a 90 per cent probability of failure ($\sigma_{90}$) at 12.19 MPa (Table 3 and Figure 3). A high value for the Weibull modulus ($m = 3.13$) was achieved when the ceramic bracket with the silane-treated base (Allure III) was bonded to the enamel using the light-cured glass ionomer cement (Photac Fil). However, the 10 per cent probability of failure ($\sigma_{10}$) was reached at 1.84 MPa and the 90 per cent probability of failure ($\sigma_{90}$) at 4.92 MPa (Table 3 and Figure 3).

The ceramic bracket with particles fused to the base (Transcend 2000) produced a Weibull modulus of 3.12 using the conventional two-paste orthodontic bonding resin (Concise). The 10 per cent probability of failure ($\sigma_{10}$) was reached at 3.10 MPa and the 90 per cent probability of failure ($\sigma_{90}$) at 8.34 MPa (Table 3 and Figure 2). When using the light-cured, fluoride-releasing orthodontic bonding resin (Sequence) and the light-cured glass ionomer cement (Photac Fil), only low values could be obtained for the Weibull modulus ($m_{\text{Seq}} = 2.68$, $m_{\text{Pho}} = 2.52$, $\sigma_{10\text{ Seq}} = 2.73$ MPa and $\sigma_{10\text{ Pho}} = 1.31$ MPa). Employing the light-cured, fluoride-releasing adhesive (Sequence), the 90 per cent probability of failure ($\sigma_{90}$) was found at 8.62 MPa. Using the light-cured glass ionomer cement (Photac Fil) the 90 per cent probability of failure ($\sigma_{90}$) was reached at 4.44 MPa (Table 3 and Figure 3).

The plastic brackets (Dentaurum Edgewise plastic bracket and Spirit bracket) produced high mean tensile strengths ($\sigma_{m\text{ Den}} = 7.23$ MPa and $\sigma_{m\text{ Spi}} = 7.36$ MPa), high bond strength values for the 10 per cent probability of failure ($\sigma_{10\text{ Den}} = 4.40$ MPa and $\sigma_{10\text{ Spi}} = 4.46$ MPa) and the highest values for the Weibull moduli ($m_{\text{Den}} = 3.77$ and $m_{\text{Spi}} = 3.74$) using a no-mix orthodontic bonding resin (System 1). The 90 per cent probabilities of failure ($\sigma_{90}$) were higher than 10 MPa ($\sigma_{90\text{ Den}} = 10.05$ MPa and $\sigma_{90\text{ Spi}} = 10.17$ MPa) (Table 3 and Figure 3).

The difference in evaluating the bond strength of a bracket-bonding system by mean values or
Figure 2  (A and B) Weibull regression lines.
by the Weibull analysis can be highlighted by the stainless steel bracket with the mesh base (Diamond) cemented to the enamel using the conventional two-paste orthodontic bonding resin (Concise) and the ceramic bracket with the silane-treated base (Allure III) cemented using a light-cured, fluoride-releasing orthodontic bonding resin (Sequence). The stainless steel bracket (Diamond) had the highest mean tensile strength in the study ($\sigma_m = 8.04$ MPa). For this
reason it would be preferable for bracket bonding to the ceramic bracket with the silane-treated base (Allure III) cemented to the enamel using a light-cured, fluoride-releasing orthodontic bonding resin (Sequence). The latter only reached an intermediate value for the mean tensile strength ($s_m = 6.43$ MPa). However, concerning the 10 per cent probability of failure ($\sigma_{10}$) the tensile stress value for the stainless steel bracket (Diamond) is only slightly higher than the value for the ceramic bracket (Allure III) (4.00 MPa compared with 3.54 MPa) (Table 3 and Figure 3). Therefore, in orthodontic treatment, comparable numbers of bond fractures can be expected for the two bracket bonding systems.

Bond failure occurred predominantly at the junction between bracket base and orthodontic bonding resin for most of the bracket-bonding systems. There were two exceptions. For the ceramic bracket with the silane-treated base (Allure III) in combination with the light-cured glass ionomer cement (Photac Fil) the bond fracture occurred both at the junction between the bracket base and the orthodontic bonding resin, and at the orthodontic bonding resin–enamel interface. There was no preferential site for the bond failure for 55 per cent of the samples. Regarding the ceramic bracket with the silane-treated base (Allure III) cemented to the enamel employing a light-cured, fluoride-releasing orthodontic bonding resin (Sequence), bracket fracture predominated with 38 per cent (Table 4). When the bond fracture was located between the orthodontic bonding resin and the enamel, enamel prisms could be identified with the scanning electron microscope and with X-ray diffraction at the orthodontic bonding resin site (Figure 4).

Discussion

The Weibull analysis is an efficient tool with which the probability of failure of bracket-bonding systems can be predicted. However, extrapolation beyond the extremes of the experimental data can lead to inaccurate predictions of the failure rates (McCabe and Walls, 1986). Although high correlation coefficients were reached for all Weibull regression lines, the lowest points of the experimental data fall well below the straight line for almost every bracket-bonding system (Table 3 and Figure 2a and b). Therefore, the clinical performance of a bracket-bonding system at low stresses may not always follow the predictions of the Weibull analysis. Outliers have to be expected at the low end of the stress range. To gain more reliable predictions, the 10 per cent probability of failure was calculated instead of the 1 per cent probability of failure used by McCabe and Carrick (1986),

Figure 4  Scanning electron micrograph of adhesive with enamel prisms (A) after tensile testing.
McCabe and Walls (1986) and Mojon et al. (1989).

The bond strength of a bracket-bonding system should be 2.9 MPa to withstand applied orthodontic forces (Miura et al., 1971; Keizer et al., 1976; Fajen et al., 1990). It should not be higher than 10 MPa, otherwise the tensile strength of the enamel may be exceeded and damage to the enamel can be caused (Bowen and Rodriguez, 1962; Diedrich, 1980; Bauer et al., 1993). The plastic brackets (Dentaurum Edgewise and Spirit) cemented with the no-mix orthodontic bonding resin (System 1) partly fulfil these requirements. For the 10 per cent probability of failure \( (\sigma_{10}) \) bond strength values higher than 4 MPa can be achieved. Moreover, the plastic brackets (Dentaurum Edgewise and Spirit) obtain the highest values for the Weibull moduli \( (m) \). This produces a close grouping of the bond strength data. However, the 90 per cent probability of failure \( (\sigma_{90}) \) exceeds 10 MPa for each of the two brackets (Table 3). Therefore, enamel failure has to be expected at debonding.

Further shortcomings are produced by the applied no-mix orthodontic bonding resin (System 1). The polymerization starts immediately after contact with the catalyst and results in rapid setting and a limited working time. Complete blending of the two phases does not occur. Moreover, no-mix materials tend to discolor and have a high polymerization shrinkage (Ferguson et al., 1984; Swartz, 1988).

Figure 5  Scanning electron micrograph showing the design of the bases of the plastic brackets (A) Dentaurum Edgewise and (B) Spirit.
The two plastic brackets (Dentaurum Edgewise and Spirit) have different base areas but the base surfaces are similar (Figure 5). For this reason the bracket with the larger base area (Dentaurum Edgewise) obtains a higher bond strength in Newtons. For example, the 10 per cent probability of failure is $\sigma_{10}^{\text{Den}} = 46.09$ N compared with $\sigma_{10}^{\text{Spi}} = 29.05$ N (Figure 6). If one takes the base areas into consideration, bond strength values are produced in MPa that are similar for the two brackets (Table 3 and Figure 3). This underlines the influence of the bracket base size on the bond strength and agrees with the findings of Buzzitta et al. (1982).

Using the light-cured glass ionomer cement (Photac Fil) the two ceramic brackets (Allure III and Transcend 2000) obtain bond strengths that are below 2 MPa for the 10 per cent probability of failure ($\sigma_{10}$) ($\sigma_{10}^{\text{All}} = 1.84$ MPa and $\sigma_{10}^{\text{Tra}} = 1.31$ MPa). The 90 per cent probability of failure is reached at 4.92 MPa for the ceramic bracket with the silane-treated base (Allure III) and at 4.44 MPa for the ceramic bracket with particles fused to the base (Transcend 2000) (Table 3 and Figure 3). Therefore, a high number of bond failures has to be expected in orthodontic treatment. The very low value of the tensile strength for the ceramic bracket with particles fused to the base (Transcend 2000) can be explained by the bracket base with its sharp-edged crystals. Cook and Youngson (1988) claim that these crystals are stress raisers which lead to bond fracture.

The presently available orthodontic brackets are designed for composite resins and are less suited for glass ionomer cement. Glass ionomer cement needs a greater thickness to achieve high tensile strengths. In thin layers glass ionomer cement tends to rupture more easily. To optimize the bond strength of glass ionomer cement, bracket designs may have to be created which produce thicker layers of the glass ionomer cement (White, 1986; Cook and Youngson, 1988; Fischer-Brandies et al., 1991; Bauer et al., 1993).

The stainless steel bracket with the mesh base (Diamond) cemented to the enamel using the conventional two-paste orthodontic bonding resin (Concise) produces a high value for the 10 per cent probability of failure ($\sigma_{10} = 4.00$ MPa). Therefore, very few bond fractures have to be

![Figure 6](image-url)  
Probability of failure in dependence of tensile stress.
expected in clinical use (Table 3 and Figure 3). Unfortunately, the 90 per cent probability of failure ($\sigma_{0.90}$) is not reached before 12.19 MPa. The consequence is the danger of enamel fracture at debonding. The value for the Weibull modulus ($m = 2.77$) does not differ significantly from the value obtained by Britton et al. (1990) for the same bracket and the same adhesive ($m = 3.4$). The characteristic level ($\sigma_0$) was substantially higher in the study by Britton et al. (1990) than in the present investigation. It reached 15.6 MPa compared with 9.02 MPa for the stainless steel bracket (Diamond). The difference can be explained by the fact that Britton et al. (1990) carried out shear instead of tensile testing.

According to the criteria used in this study it seems that the ceramic bracket with the silane-treated base (Allure III) cemented with the light-cured, fluoride-releasing orthodontic bonding resin (Sequence) is the optimum combination for bracket bonding. It obtains high values for the Weibull modulus ($m = 3.15$) and the 10 per cent probability of failure ($\sigma_{0.10} = 3.54$ MPa). The 90 per cent probability of failure ($\sigma_{0.90}$) does not exceed 10 MPa ($\sigma_{0.90} = 9.43$ MPa) (Table 3 and Figure 3). Moreover, this bracket-bonding system provides the aesthetic advantages of a ceramic bracket. Due to the fact that a light-cured orthodontic bonding resin is used, an unlimited working time is achieved.

The bond strength values for the ceramic bracket with particles fused to the base (Transcend 2000) cemented to the enamel using the conventional two-paste orthodontic bonding resin (Concise) also fall within the required limits (Table 3). However, it cannot achieve as high tensile stress values as the ceramic bracket with the silane-treated base (Allure III) using the light-cured, fluoride-releasing orthodontic bonding resin (Sequence). Moreover, the conventional two-paste orthodontic bonding resin (Concise) is less practical in use than the light-cured, fluoride-releasing resin (Sequence).

The experimental data indicate that the bond between bracket base and orthodontic resin is the weakest link in vitro. Bond fracture predominantly occurs at the interface between bracket and orthodontic bonding resin for most of the bracket-bonding systems (Table 4) (Diedrich, 1980; Eberhard et al., 1994). According to the results of Dickinson and Powers (1980), the number of bond fractures between orthodontic bonding resin and enamel will increase in vivo, because ideal bonding to enamel is much more difficult to achieve clinically. Bond failure at the enamel-adhesive interface is undesirable because of the danger of enamel fracture. This is confirmed by the finding that when bond failure occurred at the enamel-orthodontic resin interface, enamel could be identified in scanning electron microscopic examination on the adhesive site (Figure 4) (Diedrich, 1980; Diedrich et al., 1986; Swartz, 1988; Storm, 1990; Winchester, 1991).

### Table 4  Location of bond fracture.

<table>
<thead>
<tr>
<th>Bracket/adhesive</th>
<th>Bracket–adhesive interface (%)</th>
<th>Adhesive–enamel interface (%)</th>
<th>No preferential site of failure (%)</th>
<th>Bracket fracture (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diamond/Concise</td>
<td>78</td>
<td>10</td>
<td>12</td>
<td>–</td>
</tr>
<tr>
<td>Spirit bracket/System 1</td>
<td>100</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Dentaurum/System 1</td>
<td>83</td>
<td>–</td>
<td>2</td>
<td>15</td>
</tr>
<tr>
<td>Allure III/Sequence</td>
<td>33</td>
<td>13</td>
<td>15</td>
<td>38</td>
</tr>
<tr>
<td>Allure III/Concise</td>
<td>70</td>
<td>5</td>
<td>8</td>
<td>16</td>
</tr>
<tr>
<td>Transcend 2000/Concise</td>
<td>78</td>
<td>2</td>
<td>13</td>
<td>7</td>
</tr>
<tr>
<td>Transcend 2000/Sequence</td>
<td>62</td>
<td>–</td>
<td>31</td>
<td>7</td>
</tr>
<tr>
<td>Allure III/Photac Fil</td>
<td>40</td>
<td>5</td>
<td>55</td>
<td>–</td>
</tr>
<tr>
<td>Transcend 2000/Photac Fil</td>
<td>62</td>
<td>–</td>
<td>31</td>
<td>7</td>
</tr>
</tbody>
</table>
Conclusions

1. The stainless steel bracket with the mesh base (Diamond) cemented to the enamel using the conventional two-paste orthodontic bonding resin (Concise) and the plastic brackets (Dentaurum Edgewise and Spirit) cemented using a no-mix orthodontic bonding resin (System 1) achieve high bond strengths to enamel. Unfortunately, there is the danger of enamel fracture during debonding because the tensile stresses for the 90 per cent probability of failure ($\sigma_{0.90}$) exceed 10 MPa.

2. Bracket bonding with glass ionomer cement (Photac Fil) produces low tensile strength values for the 10 and 90 per cent probabilities of failure ($\sigma_{0.10} = 1.84$ MPa and $\sigma_{0.90} = 4.92$ MPa for Allure III and $\sigma_{0.10} = 1.31$ MPa and $\sigma_{0.90} = 4.44$ MPa for Transcend 2000). Therefore, a large number of bond fractures have to be expected and clinical use is not recommended.

3. The ceramic bracket with the silane-treated base (Allure III) shows a high bond strength value for the 10 per cent probability of failure ($\sigma_{0.10} = 3.54$ MPa) using the light-cured orthodontic bonding resin (Sequence), with only a few bond fractures occurring during orthodontic treatment. It can be assumed that there is little danger of enamel fracture, because the 90 per cent probability of failure ($\sigma_{0.90} = 9.43$ MPa) does not exceed 10 MPa. Therefore, this bracket-bonding system has to be especially recommended.

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