An in vitro evaluation of the tensile and shear strengths of four adhesives used in orthodontics

Patrick F. McSherry
Department of Child Dental Health, School of Dental Science, University of Dublin, Eire

Summary
The in vitro shear and tensile strength of four enamel adhesives was investigated. The materials examined were System 1+, Vitrebond, C & B Metabond and Panavia-Ex (original version). The mode of failure of each material was also investigated under a scanning electron microscope. Edgewise mesh-backed brackets were bonded to the buccal surface of extracted premolar teeth and forces applied with an Instron 1011 testing machine.

Results indicated that Panavia-Ex (with sandblasted brackets) had the highest tensile strength when compared to the other materials (P<0.01). Panavia-Ex had the highest shear strength when compared to C & B Metabond (P<0.05) and to the other materials (P<0.01). C & B Metabond had the highest shear and tensile strength of the other adhesives (P<0.01). No significant differences were found for System 1+ when compared to Vitrebond for tensile strength, but System 1+ was stronger for shear strength (P<0.01). Analysis of failure shows that 50% of the samples failed at the adhesive-enamel interface, 25% failed in a combined mode and the remaining 25% failed either adhesively to metal or cohesively.

Introduction
Acrylic or diacrylic based composite agents have gained widespread use in orthodontics, however these materials achieve a mechanical interlock to etched enamel (Reynolds, 1975). Advances in restorative dentistry have led to the development of adhesive resins which have the capacity to adhere to metal as well as etched enamel. One such adhesive, Panavia-Ex (Kuraray-Cavex, Osaka, Japan) has recently been investigated for use in orthodontics. It is a modified phosphate ester Bis-GMA composite and was introduced by Omura et al. (1984). However, Rux et al. (1991) found that the shear strength of the material was lower than a currently available adhesive (Achieve, A Company). Ireland and Sherriff (1994) found that the shear strength was greater than a no-mix adhesive (Bond Fast, Advanced Orthodontics), but after immersion in water the bond strengths were not significantly different.

Another adhesive agent, C & B Metabond (Sun Medical, Kyoto, Japan) is a trademark product based on the 4-META System. The resin consists of 4-methacryloxyethyl trimellitate anhydride. The filler consists of polymethylmethacrylate and the setting agent is tri-n-borane.

It has been found to bond reliably to enamel, non-precious metal, amalgam and unetched porcelain (Barzilay et al., 1990; Imbery et al., 1991; Cooley et al., 1992). Little research has been carried out on this material and its potential use in orthodontics.

Glass ionomer cements were introduced by Wilson and Kent in 1972. They have advantageous properties such as adhesion to enamel and fluoride release. Recently, light-cured glass ionomer has been suggested as a potential bracket adhesive. Vitrebond (3M Dental Products Division, St.Paul, MN 55144–1000, USA) is one such light-cured glass ionomer and has proved to have the highest shear strength of materials available (McCourt et al., 1991; Minnich, 1992).

System 1+ (Ormco Corp., Glendora, CA 91740, USA) is a commonly-used one paste diacrylate resin bonding agent and therefore was tested in order to compare the relative bond strengths of the other materials.

Studies on the in vitro bond strength of materials have lacked comparability, because testing methods have been different. Very few studies have tested both shear and tensile strength (Winchester, 1991). In the present
study the relative strengths of the materials were tested in both modes.

Materials and methods

Upper and lower premolar teeth (n = 80) were collected from patients having extractions for orthodontic reasons and stored in distilled water at 4°C. Standard edgewise orthodontic mesh-backed brackets were used in this study. The brackets were bonded in accordance with the manufacturer’s recommendations. Prior to bonding, each tooth was examined for any hypoplastic pits, restorations or caries and any of these teeth were excluded. The teeth were then randomly assigned for testing: 20 teeth were assigned to each of four groups. Within each group, 10 brackets were bonded to teeth used for tensile testing and 10 brackets to teeth for shear testing. Etching time was kept constant for all groups at 60 s, except the Vitrebond group which was light cured for 60 s using a Visilux light curing unit (3M Dental Products Division, St. Paul, MN 55144–1000, USA). The light source was a 75 W tungsten halogen lamp (peak power 420–500 nm). The brackets used for bonding with Panavia-Ex (original version) were sandblasted with aluminium oxide for 15 s. Sandblasting was carried out with the Basic Duo Compact Sandblaster (Renfert No. 2913 GMBH, POB 1109, D-7709, Hilzingen, Germany) with sandblasting metal type 100 (Ivoclar Vivadent, Beaumont, Leys Business Centre, Leicester LE14 1AA, UK).

After the teeth were bonded they were stored in specimen bottles filled with distilled water for 24 h at 37°C prior to testing. The teeth for shear testing were mounted in polyvinylchloride boxes using self-curing polymethyl methacrylate. A surveyor (Degussa Model No. VG3, D-6006, Frankfurt 11, Germany) was used to orientate the specimens so that the force could be applied at right angles to the tooth surface.

Tensile and shear testing were carried out using specially designed jigs on the Instron 1011 (Instron Ltd, High Wycombe, Bucks HP12 3SY, UK) (Figures 1 and 2). The instrument was calibrated after each test batch. The crosshead speed was 25 mm per min.

All samples were examined after bond failure

Figure 1 A schematic representation of the tensile testing jig.
under a scanning electron microscope (Hitachi S-520, Hitachi Scientific Instruments, Nissei Sangyo Company Ltd, Hogwood Industrial Estate, Berks RG11 4QQ, UK) at ×20 magnification. The preparation of the samples involved desiccation with silica gel for 2 days. The samples were then sputtered with gold palladium and mounted on aluminium stubs. Photomicrographs were taken with a Mamiya Camera (120 roll film back) with AGFA 120 professional film (100 ASA). The mode of failure of the bond was identified for each tooth as predominantly adhesive to metal, adhesive to enamel, combined adhesive/cohesive or cohesive. Any damage to the teeth was identified.

Statistical analysis

Descriptive statistics were calculated by using Datadesk (Version 3.0 Statistical Package for the Apple Macintosh). The tabulations are shown in Tables 1 and 3.

Tukey’s H.S.D. test was used for comparisons between the shear values and between tensile strengths for the products in the investigation.

Results

The shear strength of the materials are shown in Figure 3 and descriptive statistics in Table 1. Panavia-Ex was found to have the highest mean shear strength value of 101.5 Newtons (N) (SD 14.5 N). The minimum shear strength value of Panavia-Ex was 79.5 N and the maximum 131.75 N. C & B Metabond was found to have the next highest mean shear bond strength of 80.75 N (SD 14.6 N). The minimum value was 68.5 N and the maximum value was 107.5 N. System 1' had a mean of 55.0 N (SD 12.2 N). The minimum value was 38.5 N and the maximum value was 80.5 N. Vitrebond was found to have a mean of 35.0 N (SD 8.9 N). The minimum value was 22.5 N and the maximum value was 50.5 N.

Using Tukey’s H.S.D. test, Panavia-Ex had the highest mean shear strength when compared

<p>| Table 1 Descriptive statistics for the shear test (all measurements in Newtons). |
|---------------------------------------------------------------|-----------------|-----------------|-----------------|-----------------|</p>
<table>
<thead>
<tr>
<th>No. in sample (n)</th>
<th>Mean (x)</th>
<th>Median</th>
<th>SD</th>
<th>Min.</th>
<th>Max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>C&amp;B Metabond</td>
<td>10</td>
<td>80.8</td>
<td>75.0</td>
<td>14.7</td>
<td>68.5</td>
</tr>
<tr>
<td>Panavia-Ex</td>
<td>10</td>
<td>101.5</td>
<td>98.7</td>
<td>14.5</td>
<td>79.5</td>
</tr>
<tr>
<td>Vitrebond</td>
<td>10</td>
<td>35.0</td>
<td>33.5</td>
<td>8.9</td>
<td>22.5</td>
</tr>
<tr>
<td>System 1'</td>
<td>10</td>
<td>55.0</td>
<td>54.6</td>
<td>12.2</td>
<td>38.5</td>
</tr>
</tbody>
</table>
Distribution of the shear strength values for each of the materials in the study.

to the other materials ($P<0.01$). C & B Metabond had the highest shear bond strength of the other products studied ($P<0.01$). System 1+ was not found to have a statistically significant different mean value from Vitrebond. The results are shown in Table 2.

The tensile strengths of the materials are shown in Figure 4 and descriptive statistics are given in Table 3. Panavia-Ex had the highest mean of 137.5 N (SD 17.0 N) with a maximum value of 175.5 N and a minimum of 120.5 N. C & B Metabond was found to have the next highest tensile bond strength with a mean of 106.0 N (SD 12.0 N) with a maximum value of 77.5 N and a minimum of 135.5 N. The mean tensile strength for Vitrebond was 64.3 N (SD 12.0 N) with a minimum of 40.0 N and a maximum value of 83.5 N. System 1+ was found to have the lowest mean tensile strength of 57.8 N (SD 13.0 N) with a minimum value of 40.0 N and a maximum value of 75.0 N.

Using Tukey's H.S.D. test, Panavia-Ex was

Table 2 The values of $P$ showing the statistical significance of the difference between the means for the shear test, using Tukey's procedure.

<table>
<thead>
<tr>
<th></th>
<th>Panavia-Ex</th>
<th>C&amp;B Metabond</th>
<th>System 1+</th>
<th>Vitrebond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Panavia-Ex</td>
<td>$P&lt;0.05$</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>C&amp;B Metabond</td>
<td></td>
<td>$P&lt;0.01$</td>
<td></td>
<td></td>
</tr>
<tr>
<td>System 1+</td>
<td></td>
<td></td>
<td>$P&lt;0.01$</td>
<td></td>
</tr>
<tr>
<td>Vitrebond</td>
<td></td>
<td></td>
<td></td>
<td>$P&lt;0.01$</td>
</tr>
</tbody>
</table>

Table 3 Descriptive statistics for the tensile test (all measurements in Newtons).

<table>
<thead>
<tr>
<th></th>
<th>No. in sample ($n$)</th>
<th>Mean ($x$)</th>
<th>Median</th>
<th>SD</th>
<th>Min.</th>
<th>Max.</th>
</tr>
</thead>
<tbody>
<tr>
<td>C&amp;B Metabond</td>
<td>10</td>
<td>106.0</td>
<td>101.9</td>
<td>21.1</td>
<td>77.5</td>
<td>135.5</td>
</tr>
<tr>
<td>Panavia-Ex</td>
<td>10</td>
<td>137.2</td>
<td>131.7</td>
<td>17.0</td>
<td>120.5</td>
<td>175.2</td>
</tr>
<tr>
<td>Vitrebond</td>
<td>10</td>
<td>64.3</td>
<td>65.5</td>
<td>12.2</td>
<td>40.0</td>
<td>83.5</td>
</tr>
<tr>
<td>System 1+</td>
<td>10</td>
<td>57.9</td>
<td>55.2</td>
<td>13.1</td>
<td>40.0</td>
<td>75.0</td>
</tr>
</tbody>
</table>
found to be significantly stronger than C & B Metabond in shear testing \((P<0.05)\). C & B Metabond was the strongest of the remaining materials \((P<0.01)\). The next strongest was System 1+ and Vitrebond was the weakest (Table 4). An analysis of variance is shown for shear and tensile strength versus tooth type in Table 5.

The modes of failure for all the samples are shown in Figure 5 and those for shear testing

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**Table 4** The values of \(P\) showing the statistical significance of the difference between the means for the tensile test, using Tukey’s procedure.

<table>
<thead>
<tr>
<th>Material</th>
<th>Panavia-Ex</th>
<th>C &amp; B Metabond</th>
<th>System 1+</th>
<th>Vitrebond</th>
</tr>
</thead>
<tbody>
<tr>
<td>Panavia-Ex</td>
<td>–</td>
<td>(P&lt;0.01)</td>
<td>(P&lt;0.01)</td>
<td>(P&lt;0.01)</td>
</tr>
<tr>
<td>C&amp;B Metabond</td>
<td>–</td>
<td>–</td>
<td>(P&lt;0.01)</td>
<td>(P&lt;0.01)</td>
</tr>
<tr>
<td>System 1+</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>NS</td>
</tr>
<tr>
<td>Vitrebond</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
</tbody>
</table>

NS = not significant.

**Table 5** Analysis of variance for the shear (above) and tensile (below) strengths versus tooth type.

<table>
<thead>
<tr>
<th>Source</th>
<th>Degrees of freedom</th>
<th>Sum of squares</th>
<th>Mean square</th>
<th>(F)-ratio</th>
<th>Probability</th>
</tr>
</thead>
<tbody>
<tr>
<td>Shear strength versus tooth type</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Material</td>
<td>3</td>
<td>23721.9</td>
<td>7909.3</td>
<td>52.9</td>
<td>0.0001</td>
</tr>
<tr>
<td>Tooth type</td>
<td>1</td>
<td>538.1</td>
<td>538.1</td>
<td>3.6</td>
<td>0.0658*</td>
</tr>
<tr>
<td>Tensile strength versus tooth type</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Material</td>
<td>3</td>
<td>41600.0</td>
<td>13866.7</td>
<td>53.8</td>
<td>0.0001</td>
</tr>
<tr>
<td>Tooth type</td>
<td>1</td>
<td>486.1</td>
<td>486.1</td>
<td>1.8875</td>
<td>0.1782*</td>
</tr>
</tbody>
</table>

* Not significant.
ADHESIVE-E ADHESIVE-M COHESIVE COMBINED

Figure 5 Bar chart of the mode of failure for all the samples tested in shear and tensile.
Key Adhesive-E = adhesive to enamel.
   Adhesive-M = adhesive to metal.
   Cohesive = cohesive failure.
   Combined = combined adhesive and cohesive.

in Figure 6. System 1+ failed adhesively to enamel \((n=7)\) or combined failure \((n=3)\).
Vitrebond was seen to fail adhesively to enamel \((n=9)\) or adhesively to metal \((n=1)\).
C & B Metabond failed in a combined mode \((n=7)\) or adhesively to enamel \((n=3)\).
Panavia-Ex failed either adhesively to enamel \((n=9)\) or combined \((n=1)\).

Figure 7 shows the mode of failure for tensile testing. System 1+ was seen to fail either in a combined mode \((n=4)\) or adhesively to metal \((n=6)\).
Vitrebond failed adhesively to enamel \((n=10)\). C & B Metabond failed either adhesively to metal \((n=6)\), adhesively to enamel \((n=2)\) or combined \((n=2)\).
Panavia-Ex failed cohesively \((n=10)\). No enamel damage was found for Vitrebond, System 1+ and Panavia-Ex. C & B Metabond produced two enamel tear-outs under shear testing and two enamel tear-outs under tensile testing.

Discussion
Panavia-Ex had the highest mean tensile strength with C & B Metabond having the highest tensile strength of the remaining materials. System 1+ and Vitrebond had the lowest tensile strength. It is not possible to say with certainty that Panavia-Ex would have had the highest bond strength if the brackets had not been sandblasted. System 1+ was not found to be significantly different from Vitrebond and this result is surprising since in a previous study, Vitrebond was found to have lower bond strengths than diacrylate systems. (Rezk-Lega and Øgaard, 1991).

Panavia-Ex was found to be significantly stronger than C & B Metabond in shear testing. C & B Metabond was the strongest of the remaining materials and Vitrebond was the weakest of the materials in shear strength. Its mean after 24 h was only 35 N. This was < 58 N recommended by Tavas and Watts (1984) for clinical usage. Therefore, its potential use in areas of traumatic occlusion could be questioned.

The predominant mode of failure was adhesive to enamel. System 1+ failed in three modes, adhesive to enamel, adhesive to metal and combined failures. No trend emerged for the mode
of failure for this material. Vitrebond failed predominantly adhesive to enamel (i.e. all the material left on the bracket base). The weak link in the bonding mechanism appeared to be at the adhesive-enamel interface. Cook and Youngson (1988), and Tavas and Salem (1990) found that conditioning the enamel with polyacrylic acid had no influence on this type of failure. Sandblasting the brackets has been advocated to increase the bond strength Millett et al. (1993). It is questionable whether sandblasting the brackets in this study would have increased the bond strength because failure occurred at the enamel interface not at the metal interface.

C & B Metabond did not fail in a predictable manner. Panavia-Ex failed adhesively to enamel \((n=9)\) or cohesively \((n=11)\). Adhesive to enamel failures occurred because the sandblasting had increased the bond to the metal and because of the mode of shear testing. Cohesive failures occurred because acid etching of the enamel and sandblasting increased the bond strengths to these areas, so that failure had to occur through the cement.

Of the samples for C & B Metabond, one-
fifth (20%) had enamel fractures and this warrants further investigation, particularly when no other material had any discernible enamel damage. Finally no difference occurred in bond strength between the first and second premolars (P > 0.05) and their use together was justified.

Conclusions

1. Panavia-Ex (original version) with sandblasted brackets had the highest tensile (P < 0.01) and shear strength (P < 0.05) of the materials tested. Bond failure occurred either adhesive to enamel or cohesive.

2. Of the remaining materials C & B Metabond had the highest tensile and shear bond strength (P < 0.01). However, enamel damage occurred in 20% of the samples (four out of 20).

3. System 1+ had a statistically higher bond strength than Vitrebond (P < 0.01) in shear testing, but no significant difference occurred in tensile testing.

4. Vitrebond had the lowest shear strength of
the materials ($P<0.01$). Failure was predominantly adhesive to enamel.

5. No difference occurred between tooth types ($P>0.05$).

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References


