The Effect of Different Adhesive Systems on the Retention Strength of Bonded Amalgam Restorations

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Abstract

**Aim:** The aim of this study was to evaluate the bond strength of bonded amalgam to dentin when unfilled and filled adhesive systems are employed using different application modes and to compare the adhesives with a cavity varnish and unlined restorations.

**Methods and Materials:** One hundred twenty sound third human molar teeth were used in the study. A cylindrical cavity 3.3 mm in diameter was prepared in a cross section of dentin approximately 3.0 mm in thickness. The specimens were divided into six experimental groups (n=20) according to the cavity liner used in the prepared cylindrical cavity: One Coat Bond™ (O), Scotchbond Multi-Purpose™ (S), Panavia 21™ (Pa), PQ1™ (P), Copalite™ (C), and the unlined (U) group which served as the control group. Cavity surfaces were treated with the assigned adhesive/liner according to manufacturer’s instructions then restored with amalgam. After storage in saline solution for seven days at 37°C, the specimens were subjected to a push-out test at a crosshead speed of 1 mm/min. The mode of failure was assessed by microscopic analysis of the fracture sites. Data were analyzed by analysis of variance (ANOVA) and Duncan Multiple range tests (α=0.05).

**Results:** No significant difference in amalgam-dentin bond strength was found among O (23.47 MPa), S (21.02 MPa), and Pa (20.06 MPa) adhesive groups, but there was a significant difference between each of these groups and the P and C groups. The U group exhibited significantly lower retention than the other groups (P<0.05).
Conclusion: Different bond strengths were observed with the different types of dentin bonding agents and liners employed. The lowest bond strength was seen in the U group.

Clinical Significance: A statistically significant difference in bond strength was observed with O, S, and Pa compared to P, but this finding is not sufficient to rely on the bonding of amalgam to dentin, particularly in complex amalgam restorations.

Keywords: Bond strength, amalgam, adhesion, filled adhesives


Introduction
Decreased post-operative sensitivity and microleakage, tooth reinforcement, a reduced possibility of secondary caries, and more conservative cavity preparations are potential advantages of bonding amalgam restorations. Several in vitro studies of amalgam alloys bonding to various adhesives have shown increased retention and reduced microleakage compared to cavity varnishes. Traditional amalgam restorations are retained by preparation features that incorporate parallel or undercut walls, dovetails, box forms, and retention grooves. An additional advantage of using adhesive techniques with amalgam restorations is the increased bond strength which provides for greater structural integrity of both tooth and restoration. SEM photomicrographs showed a tooth restored with an unbonded amalgam had more spaces and artifacts at the amalgam-tooth structure interface than a tooth restored with a bonded amalgam.

Some studies apply light-cured adhesives prior to amalgam insertion, while others use chemically-cured adhesives. Winkler et al. suggested light-cured and dual-cured versions of a bonding agent provide greater retention for an amalgam restoration than does a chemical-cured bonding agent. This finding is somewhat surprising since chemical-cured liners utilize a bonding agent that sets more slowly. Therefore, the amalgam might be expected to mix with the slowly setting liner producing interlocking projections for mechanical retention. Less mixing of the amalgam and liner would seemingly occur with light-cured liners and, therefore, provide less mechanical retention. When a good retention is required, the adhesive systems beneath bonding amalgam restorations should be activated by two methods (light and chemical curing).

Bonded amalgam restorations have two important interfaces: the tooth-adhesive interface and adhesive-amalgam interface. The tooth-adhesive interface is essentially the same as that formed in bonded composite restorations and remains a matter of concern in numerous studies while the adhesive-amalgam interface has not been extensively studied. Boston has suggested chemical coupling mechanisms and mechanical intermingling of polymer and amalgam are the bonding principles. The chemical bonding between amalgam and polymer seems to be correlated with specific monomers, such as 4-META, being able to bond with metallic restorations. The mechanical intermingling of adhesives and amalgam is related to adhesive thickness and, primarily to how thick the unpolymerized resin layer is before amalgam insertion. Adhesive layer thickness is dependent...
on filler loading of the adhesive system and the method of application. While a wide variety of amalgam bonding agents and dentin adhesives generally and significantly increase retention of amalgam to tooth structure, there are differences in bond strength among these bonding agents.1

The aim of this study was to evaluate the bond strength of bonded amalgam to dentin when unfilled and filled adhesive systems are employed using different application modes and to compare the adhesives with a cavity varnish and unlined restorations.

Methods and Materials

Specimen Preparation

One hundred twenty intact and sound third human molar teeth were used in this investigation. The teeth were cleaned and disinfected in 0.5% thymol solution up to two months before use. The teeth were randomly assigned into six equal groups of 20 each for bond strength testing according to the cavity adhesive/liner used in the experiment as follows: 1. One Coat Bond™ (O) 2. Scotchbond Multi-Purpose™ (S) 3. Panavia 21™ (Pa) 4. PQ1™ (P) 5. Copalite™ (C) 6. Unlined (U)

The teeth were invested individually in clear auto polymerizing resin (Orthodontic Resin, Dentsply Caulk Co, Milford, DE, USA) and sectioned in a mesial to distal direction using a diamond saw mounted in a low-speed handpiece (KG Sorensen Ind. Com. Ltda., Barueri, SP, Brazil) to create a cross section of dentin approximately 3.0 mm in thickness. Then a 3.3 mm diameter cylindrical cavity was prepared in the dentin perpendicular to the plane of the section using a solid carbide broad point drill (D+Z, CB31L, 050606, Germany) cooled with a constant stream of water mounted in a drilling and milling machine (Model RF-20/25, Lincoln Crop, Taiwan). A jig was used to position and support the dentin specimens during the procedure to ensure parallelism of the cavity walls. Cavity surfaces were treated with each adhesive system according to the manufacturers’ instructions. The bonding systems used in this study, their characteristics, and manufacturers’ instructions are listed in Table 1.

For the C group, the dentin was rinsed, air dried, swabbed with two coats of C cavity varnish (Cooley & Cooley, Ltd, Houston, TX, USA), and allowed to air dry. The U group received no treatment.

After treatment of the prepared cavities was finished, a spherical, high copper amalgam alloy (Oralloy™, Coltene/Whaledent Inc, NY, USA) was mixed for ten seconds in an Ultramat amalgamator (Bayswater, Victoria, Australia). With one end of the prepared cavities closed with a glass slide the mixed amalgam was condensed with hand instruments and allowed to set undisturbed for ten minutes. Thirty minutes later, the samples were placed in saline solution at 37°C and stored in 100% relatively humidity chamber for seven days before being tested. All restorative procedures were performed by one trained operator.

Retention Strength Testing

The thickness and cavity diameter of the samples were measured with a micrometer caliper (LS Starrete Co, Athol, MA, USA) to facilitate a calculation of the dentin wall surface area later in the experiment. All samples were mounted in a metal jig that centered the restorations over a 4.0 mm hole with the remaining tooth structure supported by the metal sample holder. A steel rod 3 mm in diameter was centered only over the amalgam restoration and was used to apply force to the restored area of the test specimens. Specimens were tested using a hydraulically activated materials test system (Model 810, MTS Crop, St. Paul, MN, USA) (Figure 1).

![Figure 1. Schematic representation of the push-out test.](image-url)
Table 1. Components, chemical compositions, bonding procedures, and manufacturers of the bonding systems employed.

<table>
<thead>
<tr>
<th>Bonding System</th>
<th>Components</th>
<th>Chemical Compositions</th>
<th>Bonding Procedures</th>
<th>Manufacturer</th>
</tr>
</thead>
<tbody>
<tr>
<td>One Coat Bond™ (O)</td>
<td>Etchant gel</td>
<td>Water, 15% phosphoric acid, gel former</td>
<td>Etched for 15 s, rinse 5 s and lightly dried 5 s</td>
<td>Coltene/Whealedent Mahwah, NJ, USA</td>
</tr>
<tr>
<td></td>
<td>Priming Resin</td>
<td>HEMA, UDMA, Hydroxy propylmethacrylate, glycerol dimethacrylate, polyaalkenoate methacrylated, amorphous silicic acid, 5% water</td>
<td>Adhesive applied and massaged into dentin for 20 s, thin/dry 10 s Light cure 10 s - 600 mW/cm²</td>
<td></td>
</tr>
<tr>
<td>Scotchbond Multi - purpose™ (SBMP) (S)</td>
<td>Etchant gel</td>
<td>37% phosphoric acid, water</td>
<td>Etched for 15 s - rinse 15 s, gently dry 5 s</td>
<td>3M, St. Paul, MN, USA</td>
</tr>
<tr>
<td></td>
<td>Primer</td>
<td>HEMA, Polyaalkenoic copolymer, water</td>
<td>Apply one coat primer 10 s - air dry 5 s</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Adhesive</td>
<td>Bis-GMA, HEMA, Photoinitiator</td>
<td>Apply one coat adhesive-light cure 10 s - 600 mW/cm²</td>
<td></td>
</tr>
<tr>
<td>Panavia 21™ (Pa)</td>
<td>ED primer</td>
<td>ED primer; (MDP, HEMA, Water)</td>
<td>- ED Primer applied 60 s, dried with air syringe</td>
<td>Kuraray Co., LTD., Umeda, Kita-KU Osaka, Japan</td>
</tr>
<tr>
<td></td>
<td>Paste</td>
<td>Paste; (MDP, inorganic filler, comonomer, initiators)</td>
<td>- thin layer of paste applied and left “wet”</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>- amalgam restoration placed</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>- Oxyguard 11 applied to margins for four minutes prior to washing with water</td>
<td></td>
</tr>
<tr>
<td>PQ1™ (P)</td>
<td>Etchant gel</td>
<td>Water, 35% phosphoric acid, gel former</td>
<td>Etch for 15 s, rinse 5 s, Blot moist</td>
<td>Ultradent Products, South Jordan, UT, USA</td>
</tr>
<tr>
<td></td>
<td>Priming Resin</td>
<td>HEMA, methacrylic acid, phosphate monomer, ethanol, Bis - GMA, TEGDMA, filler, camphorquinone</td>
<td>Adhesive applied and rubbed into dentin 10 s, thin/dry 10 s, light cure 10 s - 600 mW/cm²</td>
<td></td>
</tr>
</tbody>
</table>

The test was done with a linear displacement mode at a cross head speed of 1.0 mm per minute. The load (N) required to push-out the amalgam was divided by the area of the cylindrical dentin surface, enclosing the amalgam and adhesive to calculate the bond strength using the following formula:

\[
BS = \frac{F}{P \times T}
\]

Where:
- BS is the Bond Strength (MPa)
- F is the Push-out Force (N)
- P is the Perimeter (mm) of the prepared cavity
- T is the thickness (mm) of the specimen.

The interfaces were examined with a stereo binocular microscope (PZO, Warsaw, Poland) at 40X magnification to evaluate the mode of failure (adhesive or cohesive). Data for each bonding system were analyzed using the analysis of variance (ANOVA) and Duncan Multiple range tests (P<0.05) to determine significant differences in retention bond strength values among the different adhesive systems.
Table 2. The mean values of retention strengths and standard deviations.

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>N</th>
<th>Mean±SD (MPa)</th>
<th>Significant Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>One Coat Bond™ (O)</td>
<td>20</td>
<td>23.47±0.04</td>
<td>No</td>
</tr>
<tr>
<td>Scotchbond MP™ (S)</td>
<td>20</td>
<td>21.02±0.03</td>
<td>No</td>
</tr>
<tr>
<td>Panavia 21™ (Pa)</td>
<td>20</td>
<td>20.06±2.02</td>
<td>No</td>
</tr>
<tr>
<td>PQ1™ (P)</td>
<td>20</td>
<td>13.15±1.04</td>
<td>No</td>
</tr>
<tr>
<td>Copalite™ (C)</td>
<td>20</td>
<td>14.14±2.01</td>
<td>No</td>
</tr>
<tr>
<td>Unlined (U)</td>
<td>20</td>
<td>4.15±1.02</td>
<td>Yes</td>
</tr>
</tbody>
</table>

Results
Retention strengths as measured by the push-out tests are shown in Table 2.

The ANOVA revealed there was a statistically significant difference among the dentin adhesives (P<0.05). The Duncan tests showed the highest bond strength values in the O, Pa, and S bonding agents. The Duncan test did not demonstrate a significant difference between the amalgam bond strength of P and C groups or among the three bonding agents (P>0.05). The U group of specimens exhibited a significantly decreased retention bond strength compared to other groups (P<0.05).

Fracture sites were evaluated for each of the adhesive systems within the study groups. Bond failures were found as follows: O (80%), S (77%), and Pa (72%). These bond failures were cohesive in nature and demonstrated remnants of adhesive materials dispersed equally between both the dentin and the restorative materials. P demonstrated an 80% adhesive failure at the amalgam-adhesive interface. The C and U groups produced fracture types that were 100% adhesive in nature with the failure located at the tooth-amalgam interface.

Discussion
Application of adhesive resins between dental surfaces and amalgam restorations in place of copal varnish has become a common procedure. There are two main reasons for using adhesives in restorative dentistry: to improve both the marginal seal and retention. In this study, light and self-cured adhesive systems were selected; because of the claims of previous studies, the mode of curing did not seem to affect the amalgam bonding. The push-out test was selected for evaluation of bond strength because this unique shear test takes a variety of stresses into consideration and better simulates the bonded dental restoration model than does flat-bonded specimens in the planner interface shear test. The development of a uniform shear stress without the presence of tensile component is an important advantage of the push-out test methodology. The push-out test in the present study was similar to the one used by Smith et al.

O is a water-based, single component, multipurpose adhesive system which can create a thick layer. This is because of the 5% filler content and the combination of primer and adhesive in a single bottle. There was no statistically significant difference between O and S in terms of retention strengths found in this study. Previous studies have shown the presence of filler content in new adhesive systems helps to create greater dentin bond strength than in dentin bonding systems without any filler because of the reinforcement of the hybrid zone.

S is also a water-based adhesive and bonds reasonably well to dentin. The retention strengths of 17-25 MPa have been reported for S. In the present study amalgam-dentin bond strength created with S was 21.02 MPa, well within the range of previous studies.

When S is used, the primer infiltrates to the etched dentin prior to resin application which promotes the adhesion of resin to dentin. The monomers in the adhesive diffuse into the dentin.

Table 2. The mean values of retention strengths and standard deviations.
forming an effective hybrid layer, which plays an important role in achieving maximum bond strength.\textsuperscript{20,21}

Pa is a self-cured resin cement commonly used as an amalgam bonding agent. It is thought Pa bonds to dentin through a self-etching mechanism, and due to its thick adhesive layer bonds reasonably well to fresh amalgam.\textsuperscript{22} The results of this study were consistent with the findings of Winkler et al.\textsuperscript{9} who recognized Pa significantly increased the retention of amalgam to tooth structure. The high film thickness along with the interlocking of amalgam-Pa and dentin could be the reasons for a higher amalgam dentin bond strength.\textsuperscript{3} The concern regarding the use of a chemical-cured version of the adhesive system is the likely inclusion of resinous material within the amalgam body.\textsuperscript{10} Some studies have already reported a reduction in the mechanical properties of amalgam under this situation.\textsuperscript{23}

Yoshida et al.\textsuperscript{24} reported adhesive monomer bonded to the hydroxyapatite and formed a calcium-monomer salt. The bonding efficacy of the adhesive monomer was determined by the velocity of the chemical reaction and stability of the salts. The 10-metharyloxy decyl dihydrogen phosphate (10-MDP), which was introduced in Pa, exhibited high efficacy as the bonding monomer. The 10-MDP rapidly forms a stable salt with the dentin calcium compared to other monomers such as 4META, Phenyl p, which is comparable to carboxyl groups in the polyalkenoate methacrylated one found in O and S.\textsuperscript{21}

P is also a viscous, single component, multipurpose adhesive system. Its solvent is ethyl alcohol with 40% filler loading. In the present study the retention strength of amalgam was statistically less than the other adhesives when P was used. This is probably due to higher filler loading of P, type of solvent, and the functional monomers. The higher filler loading of P, the greater viscosity of the bonding system. The type of solvent (alcohol) which evaporates more rapidly than water could prevent optimal adaptation of adhesive to the etched dentin and the exposed collagen fibers. As a result, a suitable hybrid layer may not form.\textsuperscript{25} Therefore, it seems the threshold of filler loading into the dentin bonding agent is an important factor for creating a suitable bond strength. If PQ™ amalgam adhesive had been used in this study instead of P, a higher bond strength might have been obtained. In this study there was no significant differences among the retention strengths of O, S and Pa.

It should be noted an increase of 10 MPa in retention strength between O and P will not be sufficient for a reliable bond from amalgam to dentin. Selecting an adhesive material based on the results of tests performed in vitro requires careful consideration of the requirements of the specific clinical application. A complex amalgam may require high bond strength values to facilitate retention.\textsuperscript{2} The true significance of the amalgam bonding technique may be in its superior sealing efficacy, particularly when spherical alloys are utilized. Increased caries resistance around amalgam bonded restorations has even been suggested.\textsuperscript{26}

Oralloy™ is a spherical, high copper amalgam alloy that contracts on setting.\textsuperscript{27} If another brand of amalgam is used that does not contract on setting, a greater setting expansion will result in a greater wedging effect of the alloy against the cavity wall, thereby, enhancing the retention of the restoration.\textsuperscript{28}

In the resin bonded amalgam the weakest link is the amalgam/resin interface due to the inherent stiffness of amalgam.\textsuperscript{29} The fracture site results of this study suggest the highest bond strengths occurred when a majority of failures were cohesive in nature and the weakest bonds were associated with a high percentage of adhesive failures. It is generally accepted the attachment mechanism is achieved largely by the intermingling of adhesive resin and unset amalgam at the time of amalgam insertion.\textsuperscript{21} The attachment between O, S, and Pa with the hybridized dentin surface was greater than between the adhesive resin and amalgam. For most adhesive systems, residual resin liner was usually found on the dentin walls of preparations after testing to the level of failure and little, if any, resin remained on the amalgam. Thus, improving the cohesive failure characteristics of bonding systems holds the potential for enhancing the bond between the amalgam and bonding agent. These results were consistent with previous studies.\textsuperscript{2,9,29}
Further research must be done to verify the retention strength and SEM observations of different bonding agents when used for amalgam bonding in association with different types of amalgam alloys.

Conclusions
Considering the limitations of this study, the following conclusions could be drawn:

1. Filled adhesives can produce dentin bond strengths greater or lesser than those of unfilled adhesives. Therefore, the addition of filler to an adhesive is not a significant determinant of adhesive bond strength.

References

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