# The bonding of composite resin to moist enamel

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**Objective** To determine the effect on the bond strength of modern dentine bonding agents to etched enamel of surface contamination with water.
**Design** Fifteen bond strength measurements were made for bonds prepared to both moist and dry etched enamel for each of three test and one control materials.
**Results** For two materials (Scotchbond 1 and Prime and Bond 2.1) the bond strength was not affected by the presence of water on the etched enamel surface. A mean bond strength in excess of 25 MPa was achieved for both materials under all conditions. One material (Optibond Solo) showed a 30% increase in bond strength when bonds were formed under wet condition (21.10 MPa compared with 15.35 MPa). The bond strength of the control material, a conventional unfilled bonding resin, decreased markedly with aqueous contamination (9.14 MPa compared with 26.75 MPa).
**Conclusion** Etched enamel should be rehydrated routinely prior to bonding composite resin to its surface using a water displacing dentine bonding system.

The ability to bond composite resin reliably to enamel forms the basis of much of adhesive dentistry. The technique was pioneered by Buonocore,[1] and was made a practical reality with the development of modern composite resins using Bis GMA or other long-chain dimethacrylate resins as their basis. The technique comprises the pretreatment of an enamel surface with acid to render it microporous and the subsequent infiltration of the porous surface with a low viscosity resin.[2–4] This produces a micromechanical bond of considerable strength. The 'bonding resin' for early composite materials comprised simply of the binding resin of the composite (ie the dimethacrylate resin itself) which may also have contained some diluents to improve flow. It was established at a relatively early stage in the evolution of this technique that contamination of the dried etched enamel surface with water, saliva or GCF resulted in failure of resin penetration and dramatic reductions in bond strength.[5–8]

Developments in bonding resins have moved us away from simple dimethacrylate resins to complex and chemically active resin systems designed principally to bond to dentine, rather than to enamel. Once again, these resins perform well when bonded to dry etched enamel.[9–11] However, the most recent concepts in dentine bonding demand that the etched dentine surface is fully hydrated to permit maximal penetration of the resin into the exposed collagen network, thus forming the 'hybrid layer'. In a clinical setting it is not possible to produce damp dentine with dry etched enamel around it.

There are few data in the literature showing the effects of bonding modern adhesive resin systems to damp etched enamel, compared with dry etched enamel. The data that are available give some confusing results with some authors reporting reduced bond strengths,[11,12] no effect,[13] or improved bonding.[9,14–16] Some of these data relate to bonding systems that are no longer available. Furthermore, there is limited evidence of the effect of reducing the numbers of 'steps' involved in a bonding protocol on bond strength to enamel.

The aim of this study was to compare the bond strengths in shear of three modern dentine bonding systems to etched enamel that was either dry or moist at the time of application of resin. A 'classical' unfilled dimethacrylate resin was used as a control.

Our null hypothesis was that the presence of moisture would have an adverse effect on the bonding of resins to etched enamel.

## Materials and methods

The bonding resins used are described in Table 1. Sixty recently extracted human premolar and molar teeth were used as the enamel source for this study. The teeth were inspected under binocular magnification to ensure that the enamel was grossly intact, cleared of debris and stored in 0.5% chloramine solution at 4°C for 4 weeks before being transferred to distilled water until specimen preparation.

The buccal surface of each tooth was ground on a rotary pregrinder using 250 and 500 grit silicon carbide paper under water cooling until a flat enamel surface with a minimum diameter of 5 mm was produced. These teeth were then embedded in cylindrical polyester resin blocks with the ground surface in the centre of and parallel to the flat end of the resin block. When the mounting resin had cured fully, the blocks were placed in an alignment jig and final preparation of the enamel surface was undertaken with 800 grit silicon carbide paper. All completed specimens were stored in distilled water at 4°C prior to use

Size 5 hard transparent gelatine capsules, designed for pharmaceutical applications, were used as matrices for the production of columns of composite bonded to the enamel surface. The capsule was part filled with a standard composite material to limit the incremental thickness of the bonded material to 1.5 mm. A self-adhesive washer with a central hole 4.5 mm in diameter was applied to the enamel surface. The enamel surface was then etched, for the manufacturer's recommended time, with the acidic conditioner appropriate for each system. After etching the enamel surfaces were washed with water for 15 seconds and dried with oil-free air until the typical chalk-like appearance of etched enamel was observed.

For those specimens that were scheduled to have wet enamel surfaces, the enamel was rehydrated using the technique suggested by Powers *et al.*[16] The dried enamel surface was moistened thoroughly using tap water and a small sponge applicator. This left a surface obviously saturated with fluid. Excess water was then removed by blotting the surface with a cotton pledget to leave a damp etched surface similar to that which would be encountered clinically.

The bonding procedures to both wet and dry enamel were then

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performed following the manufacturer's instructions. The sixty tooth specimens were divided at random into four groups of fifteen and each group was assigned to a material. Two bonds with the same material were made to each tooth surface, one wet and one dry. Half of the surfaces were bonded wet during the first testing round and dry during the second and vice versa for the remainder. The enamel surface was re-prepared with 800 grit silicon carbide paper between the two bond strength tests.

All specimens were stored at 37°C and 100% relative humidity for 24-hours between preparation and bond strength testing. The bond strengths were determined in shear using an Instron 5567 Universal testing machine in a shear testing jig that conforms to ISO TR11405. Testing was carried out at a crosshead speed of 0.5 mm/minute.

Following testing to failure, the enamel surfaces were examined under a binocular microscope at 4.5 magnification to determine the mode of fracture.

## Statistical analysis

Student *t*-tests were used to determine the effects of wet or dry enamel surfaces for each material. A one-way analysis of variance (ANOVA) was used to determine if there were differences between materials bonded to wet or to dry surfaces. When the ANOVA proved significant Tukey's multiple comparison test was used to determine the nature of the differences. Weibull analysis was used to determine the relationship between probability of failure and stress in each case.

## Results

The bond strength data are given in Table 2.

The control material showed a marked and significant reduction in bond strength when applied to a surface contaminated with water ($P < 0.01$). Scotchbond 1 and Prime and Bond 2.1 showed no differences between wet and dry surfaces ($P > 0.05$). Optibond Solo demonstrated a greater bond strength to wet enamel compared with that to dry enamel ($P < 0.05$).

When a between materials comparison was made for the different bonding conditions, there was no difference between the control, Scotchbond 1 and Prime and Bond 2.1 on dry enamel. Optibond Solo showed a significantly lower bond strength than the other two materials (($P < 0.05$) Tukey's multiple comparison). For wet enamel, there was no difference between Scotchbond 1 and Prime and Bond 2.1. Optibond Solo exhibited stronger bonds than the control but weaker than the other two bonding systems (($P < 0.05$) Tukey's multiple comparison).

The results of the Weibull analysis are given in Table 3. The control material Margin Bond showed a marked reduction in bond reliability when the enamel surface was contaminated with water. Between 80 and 100% of such bonds would have failed at an applied stress of 17 MPa. The reverse was true for Optibond Solo and, to an extent, Scotchbond where bonds formed to dry enamel were less reliable at the critical stress of 17 MPa than those formed to damp enamel. The bonds formed by Margin Bond to dry enamel, Optibond Solo and Scotchbond to wet enamel and Prime and Bond to both wet and dry enamel showed similar reliability. However, those formed by Optibond Solo did demonstrate a lower characteristic stress.

All materials showed mixed adhesive/cohesive failure on inspection with the exception of the control material bonded to wet enamel. In addition, there was some evidence of physical damage to the enamel surface for both Scotchbond 1 and Prime and Bond 2.1 under both conditions of bond formation.

## Discussion

The enamel specimens in this study were each used for two bond strength tests with the surface of the enamel being refinished with 800 grit wet and dry paper between tests. The objective of the refinishing process was to remove any residual resin tags from within the used enamel surface. Resin tags have been reported as penetrating up to 100 µm into etched enamel. Whilst we did not measure the extent of enamel removed by the refinishing procedure for the enamel surfaces, the bond surface was re-prepared using abrasive paper to give a freshened smooth enamel surface for etching. Each etched surface was examined visually, with care, to ensure that no islands of resin were remaining (these would manifest themselves as areas where the etched dried enamel would not have its characteristic chalk-like appearance). Finally, our pilot studies showed that there were no differences between the two bond strengths obtained for each specimen using this protocol. Assignment of materials to enamel specimens and whether the enamel surface was being used for the first or second time were undertaken on a random basis to minimise any potential bias.

The measured bond between enamel and resin serves two purposes, to help to retain the restoration in place and to help to resist the forces of polymerisation shrinkage and hence micro-leakage. It has been estimated that a shear bond strength of the order of 17 to 20 MPa is required to achieve these objectives.[17,18] The bond strengths developed by the materials in this study were, for the most part, in excess of these figures. However those for the control material to wet enamel (9.21 MPa) and Optibond Solo to dry enamel (15.35 MPa) were below this level. The reliability of bonds formed by the control material to wet enamel and Optibond Solo to dry enamel was also poor, with high probability of bond failure. There was also a trend for bonds formed by Scotchbond 1 under dry conditions, to be less reliable than those formed under wet conditions despite similar mean bond strength and characteristic stress.

These data support previous findings that surface contamination with a conventional resin bonding system results in a serious

**Table 2 Mean bond strengths of composite resins to etched enamel prepared perpendicular to the enamel prism axis (Standard deviations are given in parenthesis)**

| | Dry | | Wet | |
|---|---|---|---|---|
| Scotchbond 1 | $26.21^a$ | (10.06) | $31.07^a$ | (4.81) |
| Prime Bond 2.1 | $29.29^a$ | (3.91) | $27.13^a$ | (5.24) |
| Optibond Solo | $15.35^b$ | (4.74) | $21.81^b$ | (3.16) |
| Control (Margin Bond) | $26.75^a$ | (4.80) | $9.13^c$ | (5.71) |

The horizontal lines indicate no difference within the material between surface treatments.
The superscripts (a,b and c) denote no difference between materials from a given surface treatment

**Table 1 The materials used in this study**

| Adhesive resin | Batch number | Composite | Batch number | Manufacturer |
|---|---|---|---|---|
| Scotchbond 1 | 19970408 | Z-100 | 7BY2000-11 | 3M |
| Prime Bond 2.1 | 9710000984 | Spectrum TPH | 9802001259 | Dentsply |
| Optibond Solo | 705455 | Prodigy | 503716 | Kerr |
| Control (Margin Bond) | FA367 | Brilliant | 9209159 | Coltene |

**Table 3 Weibull parameters and predictions of failure for the bonds between resins and both dry and damp enamel**

| Adhesive | Surface condition | Characteristic stress (Mpa) | Weibull modulus | 95% confidence intervals for failure at 17 MPa |
|---|---|---|---|---|
| Margin Bond | Dry | 28.7 | 5.1 | 0–10 |
| | Wet | 10.2 | 1.7 | 80–100 |
| Scotchbond 1 | Dry | 29.3 | 3.0 | 4–30 |
| | Wet | 33.1 | 7.1 | 0–3 |
| Prime and Bond | Dry | 30.9 | 10.0 | 0-2 |
| | Wet | 29.2 | 6.1 | 0–10 |
| OptiBond | Dry | 16.7 | 3.9 | 50–80 |
| | Wet | 23.0 | 10.8 | 0–10 |

diminution in bond strength that would be liable to lead to early and catastrophic failure of the restoration.

The performance of Optibond Solo is somewhat puzzling. This system contains a resin primer/adhesive dissolved in ethanol with some admixed water. Previous experience would suggest that this combination would facilitate penetration of the resin into dry etched enamel and yet these data do not support this with a poor measured bond strength. One explanation may be that this material requires the presence of moisture for effective penetration of such a high energy surface. This contention is supported by an increase in bond strength of the order of 30% under wet conditions. Scotchbond 1 and Prime and Bond 2.1 performed equally well in both wet and dry environments.

The problems of displacing water from the porous surface of etched enamel are greater than those for dentine. Etched enamel is a very high-energy surface that will bind water effectively and firmly. These dentine bonding systems are obviously capable of either displacing this tightly bound water or of combining with it within the porous surface to produce an effective bond and displace any free water from the etched surfaces.

## Clinical implications

Within the limits of this study, it would appear that bonding to wet enamel should not be a problem clinically with modern, water chasing, dentine bonding agents. Indeed with one of the products tested the bond strength to moist enamel was greater than that to dry enamel. Consequently, we would recommend the routine rehydration of enamel if it has been desiccated during etching, washing and drying prior to the application of a dentine bonding agent on enamel.

When a classical unfilled resin system is being used, it remains essential that a clean, dry etched enamel surface is prepared. Aqueous contamination of the etched enamel with such a resin resulted in a 66% drop in bond strength. This would result in early and catastrophic failure of bonds if reproduced clinically.

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