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Investigation of a Hydrophilic Primer for Orthodontic Bonding: an *in vitro* study

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Abstract. *A common reason for bond failure is moisture contamination. This study investigates the* in vitro *bond strength of brackets bonded using a new hydrophilic primer, designed to be insensitive to moisture, and compares it with a conventional primer. Using a standardized technique, the* in vitro *bond strength of brackets bonded with the hydrophilic primer was compared to identical brackets bonded with a conventional primer. Although designed to be moisture insensitive, the directions for use stipulate drying the teeth before bonding. Therefore, for the purposes of comparison with a conventional primer the experiment was conducted under dry conditions. The results were analysed using the Weibull distribution modelling.*

The median bond strength with the hydrophilic primer (6·43 MPa, 95 per cent C.I. 7·69–9·50) was significantly lower (P = *0·0001) than the conventional primer (8·71 MPa, 95 per cent C.I. 5·89–7·59). The Weibull distribution modelling showed that brackets bonded with the hydrophilic primer were 3·96 times more at risk of failure (95 per cent C.I.: 2·39–6·56;* P *<0·0001). The bond strength at which 5 per cent of the brackets failed was also lower for the hydrophilic primer.*

The bond strengths obtained with the hydrophilic primer were significantly lower than with the conventional primer. Although the median bond strength values were promising, the laboratory results for this particular hydrophilic primer were disappointing when using the Weibull analysis, where the whole distribution of bond strength is taken into account.

Index words: Hydrophilic Primer, Moisture Insensitive Primer, Orthodontic Bonding, Weibull Analysis.

Introduction

Attaching orthodontic appliances directly to enamel using an acid-etched technique was first performed in 1965 (Newman, 1965). This process is colloquially known as 'bonding'. It is a technique sensitive procedure which, if performed incorrectly, can lead to an increase in bond failures. One of the commonest reasons cited for bond failure, particularly on posterior teeth, is moisture contamination (Wertz, 1980; Kinch *et al.*, 1988; Wang and Lu, 1991). This can occur despite attempts at stringent moisture control. It would therefore be helpful, as part of the bonding procedure, to incorporate a feature which overcomes this.

The majority of current adhesives used for orthodontic bonding are composite resins based on the bis-GMA formula (Turner, 1996). There has been much interest in the use of glass ionomer cements or glass polyalkenoates, which have, among other favourable properties, a tolerance of moisture. However, both *in vitro* and *in vivo* studies indicate that conventional glass ionomer cements are unreliable for clinical orthodontic bonding. Hybrid materials composed of glass ionomer and resin components appear to have a greater potential with regard to clinical performance, but further developments are required before they can be recommended for routine clinical use (Millett and McCabe, 1996).

The effect of moisture contamination on bond strength of composite to enamel has been thoroughly investigated. Hormati *et al.* (1980) looked at the effect of saliva contamination on the quality of acid-etched enamel and its effect on shear bond strength. They showed shear strength reduced by 50 per cent in the presence of moisture and that simply drying off the saliva was not sufficient. Scanning electron micrographs demonstrated an etched enamel pattern with porosities plugged by moisture, so the depths of the composite tags were of insufficient depth for adequate retention. Even a momentary contamination with saliva has been shown to adversely affect the bond (Silverstone *et al.*, 1985). When exposed to saliva for a second or more, the etched enamel is coated with a tenacious surface coating that cannot be removed by simple washing.

The problem of moisture contamination has been addressed in the development of dentine bonding agents. Dentine is 51 per cent organic material and water (Vadiakas

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and Oulis, 1994), so is an inherently moist surface. The bulk of dentine bonding agents are bi- or multi-functional molecules, which have reactive groups that interact with the monomer of the resin, and other groups that react with the organic and/or the inorganic components of dentine. Third generation dentine bonding agents were developed with a hydrophilic component, such as hydroxyethyl methacrylate (HEMA), which allows a lower contact angle with the prepared dentine and an extension of the molecule that will readily bond to the resin composite (Thoms *et al.*, 1994). When dissolved in an acetone solvent, which is highly miscible with water, it is even more effective (Jacobsen and Söderhom, 1995).

The use of dentine bonding agents containing hydrophilic primers when bonding to enamel has also been investigated. Using the HEMA-based 'Scotchbond Multi-Purpose', equivalent bond strengths to enamel were achieved in dry or moist conditions (Vargas *et al.*, 1994). In work aimed more specifically at the bonding of orthodontic brackets to moist enamel results have also been encouraging. Using 'Scotchbond Multi-Purpose' *in vitro* the bond strengths of brackets bonded to saliva-contaminated and uncontaminated teeth were equivalent (Sonis, 1994). Using the same bonding system Ibe and Segner (1995) showed similarly successful results with enamel contaminated with saliva and blood. They also pointed out that there was no negative effect on bonding in the dry state.

A new hydrophilic primer, compatible with composite resin, has been developed by 3M Unitek specifically for use in orthodontics. It contains a hydrophilic primer (HEMA and malic acid) dissolved in acetone.

This *in vitro* study investigated the bond strength of brackets bonded with the new hydrophilic primer and compared it with brackets bonded using a conventional primer.

Materials and Methods

Teeth

Extracted human sound premolar teeth were collected from patients under 18 years old and soft tissue remnants removed. They were stored in 0·5 per cent aqueous chloramine-T solution at 4°C, as a decontaminant, for 1 week. They were then stored in distilled water at 4°C from between 1 and 6 months. The roots were grooved with a diamond bur to aid retention following mounting.

Brackets

Adhesive pre-coated (APC) metallic Mini Uni-Twin® (3M Unitek, Bradford, U.K.) 0·022-inch pre-adjusted Edgewise upper premolar brackets were used. A consistent quantity and quality of composite resin is placed on the bracket by the manufacturer. This reduces unwanted bonding variables introduced by inconsistencies in the adhesive.

Bonding System

Two types of primer were used:

1. New hydrophilic primer supplied by 3M Unitek.
2. Conventional Transbond adhesive primer (unfilled compatible resin) to act as a control.

An Ortholux XT® (3M Unitek) visible light-curing unit was used for polymerization.

Sample Size

Thirty specimens per test group were used. This gives approximately 80 per cent power to detect a statistically significant difference between the two primers ($P < 0{\cdot}05$), based on a Mann–Whitney test, if there really is a difference of 1 MPa.

Bonding Procedure for Each Tooth

For each type of primer 30 teeth were bonded at an ambient temperature of 24°C using the following protocol:

1. Oil-free prophylaxis.
2. Thirty-second wash and 30-second dry using 3-in-1 syringe.
3. Thirty-second etch with 37 per cent phosphoric acid gel.
4. Thirty-second wash and 30-second dry using a 3-in-1 syringe.
5. Application of relevant primer to acid-etched enamel and air thin.
6. APC bracket placed at long axis point on buccal surface of tooth.
7. Light polymerization: 30-second mesially and distally of each bracket.

Subsequent to bonding the samples were stored in distilled water at the physiological temperature (37 °C) for 24 hours, prior to testing.

Bond Strength Testing

In order to maintain a consistent debonding force in a controlled direction a mounting jig was used to mount the teeth in acrylic in the same plane (Figure 1). A second debonding jig was then used to debond the brackets exactly perpendicular to this plane (Littlewood and Redhead, 1998). The force to debond was recorded with a universal testing machine (Lloyd® Instruments, Fareham, U.K.: NAMAS certified No. 980108). A crosshead speed of 0·5 mm/min was used.

As the universal testing machine simply records the 'force to debond', the bond strength was calculated by dividing this figure (in Newtons) by the area of the bracket base (8·3 mm^2). The measurements were made with electronic digital callipers. These callipers were calibrated with stainless steel calibration blocks and the measurements repeated 1 week apart to check for reproducibility. The bracket base area is only approximate as the bracket is compound contoured.

Site of Bond Failure

This was assessed under ×40 magnification and graded according to the Adhesive Remnant Index or ARI (Årtun and Bergland, 1984).

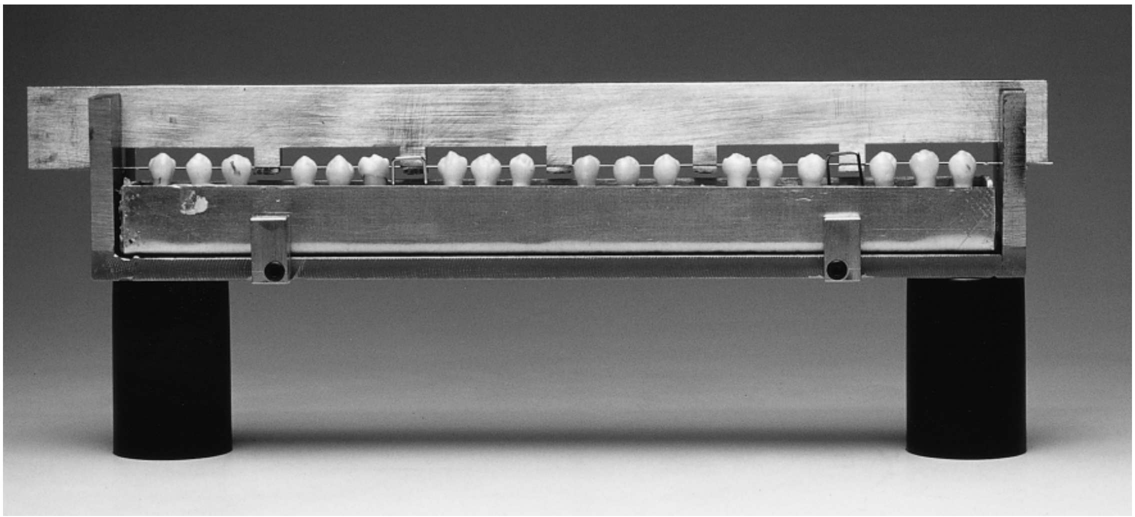

FIG. 1 Photograph of teeth on mounting jig.

Statistical Analysis

The mean bond strength quoted in many studies would exceed the bond strength that has been recommended to resist clinical failure (Reynolds, 1975). Hence, when considering bond failure, the whole spread of the data is important, particularly the weaker values (the lower tail of the distribution). The Weibull analysis, a survival analysis first described in 1951 by Weibull, is ideal for analysing this type of data that may not be normally distributed (McCabe and Carrick, 1986). This is because it models the tails of the distribution. It is used in the evaluation of biomaterials and devices, and has been suggested as an appropriate test for orthodontic bond testing (Fox *et al*., 1991, 1994; Fox and McCabe, 1992; Millet and McCabe, 1996).

To allow statistical comparison of the two primers a modelling approach to calculating the Weibull modulus was performed using Stata (Stata Corp, 1997) and SAS version 6.12, PROC LIFEREG (SAS Institute, 1989) and confirmed using GLIM 4 (Aitkin *et al*., 1989; Francis *et al*., 1993). Separate models were constructed for each primer to estimate the Weibull modulus and normalizing parameter (characteristic strength). To compare primers directly a combined model was used, with an extra term for the different effects of the primers.

As it is the lower values of bond failure in which we are interested, the stress for 1 and 5 per cent chance of failure were also calculated from the Weibull models, and presented with 95 per cent confidence intervals.

To distinguish between the primers hazard ratios were calculated. These present the probability of failure of the primers as a ratio. This provides a means of relating the risk of failure of one primer to another when considering the whole distribution. Where appropriate, approximate 95 per cent confidence intervals were calculated.

Results

Basic Bond Strength Data

Median values were calculated and the 95 per cent confidence interval for the median quoted (see Table 1). Using the Mann–Whitney *U*-test there is a statistically significant difference between the medians (P = 0·0001). The 95 per cent confidence interval for the difference between the population medians is 0·96–2·77.

TABLE 1 *Basic descriptive statistics of bond strength data (MPa)*

Primer type	Median	Max.	Min.	95% CI of median
Conventional primer	8·71	12·26	5·05	7·69–9·50
Hydrophilic primer	6·43	9·01	3·85	5·89–7·57

The results of Adhesive Remnant Index (ARI) data indicated that there was no association with primer type (chi-squared test: χ^2 = 0·5 on 2 degrees of freedom, P = 0·78).

Weibull Analysis

The Weibull distributions for each type of primer are shown graphically in Figure 2. The Weibull modulus and characteristic strength were calculated using Weibull distribution

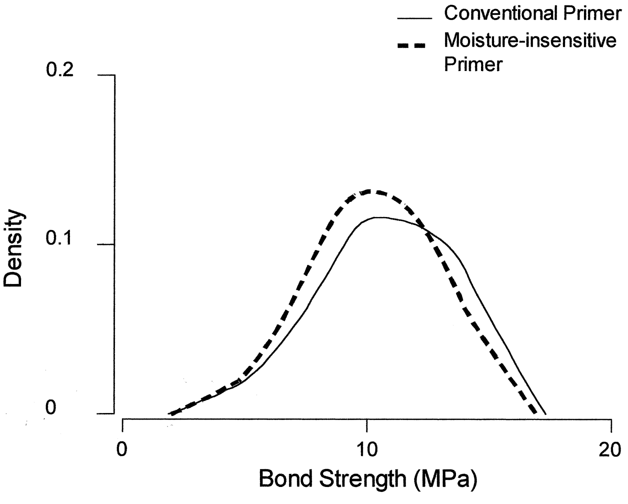

FIG. 2 Graphs of Weibull distributions for each primer.

TABLE 2 *Comparison of primers using Weibull distribution modelling*

Initial bonds	Weibull modulus (95% CI)	Characteristic strength (95% CI)	Stress for 1% chance of failure (MPa, 95% CI)	Stress for 5% chance of failure (MPa, 95% CI)
Conventional primer	5·36 (4·20–7·40)	11·98 (11·62–12·37)	4·0 (3·0–5·2)	5·4 (4·5–6·5)
Hydrophilic primer	5·60 (4·36–7·83)	11·14 (10·78–11·50)	3·2 (2·5–4·2)	4·3 (3·6–5·2)

modelling. This allowed statistical comparisons of the Weibull distributions. As the lower values of bond failure are of greater importance clinically, the stress for 1 and 5 per cent chance of failure were also calculated from the Weibull models, and presented with 95 per cent confidence intervals (see Table 2).

Brackets bonded with the hydrophilic primer were almost four times more likely to fail than brackets bonded with the conventional primer (hazard ratio = 3·96; 95 per cent CI: 2·39–6·56; *P* <0·0001).

Discussion

This *in vitro* study has shown that brackets bonded with the new hydrophilic primer have a lower bond strength than those bonded with a conventional primer. Whether these results can be extrapolated to the clinical situation depends on how appropriate and representative the laboratory test conditions are.

Laboratory bond testing is designed to evaluate the bond strength of adhesives to provide an indication of the risk of clinical bracket failure. The approach used should be simple enough to be reproducible, but ideally sophisticated enough to be valid.

The methodology used in this study was largely based on a protocol designed to increase the reproducibility of bond strength testing (Fox *et al*., 1994). One area of this protocol that was not consistently reproducible in previous studies was the exact direction of the force used to debond the brackets. This problem was addressed by using jigs to mount and debond the brackets (Littlewood and Redhead, 1998). This attempted to restrict the debonding force to pure shear, minimizing the unwanted and unpredictable affects of peel. To improve reproducibility in any future research it would therefore be advisable to carefully control the direction of the debonding force.

Validity of *in vitro* bond strength testing requires the laboratory techniques to be truly representative of the debonding forces acting clinically. This study could be criticized for not fully recreating the oral conditions *in vitro*. However, this is a very complex area as clinically bonded brackets are subjected to a whole range of different forces acting at different temperatures in different levels of humidity. The forces may dislodge the brackets in single traumatic incidents (comparable to the universal debonding machine *in vitro*) or as a result of repeated stresses (Zachrisson *et al*., 1996). Various workers have tried to reproduce these clinical conditions in the laboratory with cyclic stressing (Moseley *et al*., 1995), use of a ball mill to introduce varied forces (Abu-Kasim *et al*., 1996) and thermocycling (Zachrisson *et al*., 1996). At this time, there is no good evidence that these approaches, although theoretically sound, are appropriate. The closer *in vitro* bond testing tries to mimic the clinical situation the more complex it becomes. The increase in complexity often compromises reproducibility. If it can be shown that simple additions to the *in vitro* bond testing protocol increases the validity then these may be introduced in the future.

In this study, the APC brackets were used as they provided control of bonding variables, with a consistent quality and quantity of composite resin adhesive. This control of bonding variables is particularly useful when studying other aspects of the bonding procedure—such as the effect of primers. This is because any changes in the bond strength are a reflection of the independent variable being tested, rather than due to inconsistencies in the technique. Ideally, a standard bracket should be used for *in vitro* bond strength testing as this would permit more valid comparisons between studies.

One of the possible shortcomings of this study is that although a hydrophilic primer was being investigated, all the tests were done under dry conditions. This was because of the difficulty in standardizing not only the amount of moisture to use, but also the type.

Although many primers have been designed to bond in moist conditions it is possible for excess water to compromise the bond (Tay *et al*., 1995). It would therefore be difficult (if not impossible) to determine how wet to leave the tooth surface. For the research to be scientifically valid the quantity of moisture present on every tooth would have to be reproducibly accurate. As well as the quantity of moisture present the type of moisture is important. In the clinical situation the moisture present could be residual water left after etching and washing the teeth. However, moisture contamination could be due to oral fluids such as saliva, gingival exudate, or even blood. It would ideally be necessary to test the primers in the presence of water, blood, saliva, and gingival exudate. The matter is complicated further by the fact that saliva and gingival exudate can differ greatly in composition according to the situation under which they are produced.

The premise for testing under dry conditions was therefore that it provided the most scientifically controlled approach. The manufacturers stipulate in the instructions for use of the new primer that a normal bonding protocol of drying the teeth before placing the primer is advisable. A primer with a similar chemical composition to the hydrophilic primer has previously been shown to bond to enamel equally well in wet and dry conditions (Ibe and Segner, 1995). In this laboratory study, where dry conditions were guaranteed, there should have been no difference in the results between the two primers.

It could be argued that the presence of water may have improved bond strengths with the hydrophilic primer. Certainly, some dentine bonding agents have been shown to be more effective in the presence of water, but this is less likely to happen with enamel bonding because enamel structure is radically different from dentine. One of the principle advantages of water in dentine bonding is that it helps to maintain the collagen framework into which the monomers penetrate to provide micro-mechanical retention. If dentine becomes dehydrated the collagen network collapses and the opportunity for micro-mechanical retention is severely reduced (Pashley & Carvalho, 1997). When enamel is dehydrated the etched surface is not compromised and the opportunity for micro-mechanical retention is maximized.

Products that can bond to wet dentine may provide ideas for the development of primers for bonding to wet enamel. However, it is important to recognize that since the structure of dentine and enamel is so radically different what may be successful in bonding to wet dentine may not be appropriate for bonding to wet enamel.

Many previous studies have simply described the Weibull distributions in terms of the Weibull modulus and the characteristic strength, without attempting to formally test for differences between these distributions (Fox and McCabe, 1992; Bearn *et al.*, 1995; Nkenke *et al.*, 1997). This study has tested for a statistical difference between the Weibull distributions of the bond strengths of each primer using the hazard ratio.

The diversity of materials and methods used makes comparisons of different data almost impossible (Rueggeberg, 1991). Although more recent studies often follow a more standardized protocol (Fox *et al.*, 1994) a comparison of data between studies should be interpreted with caution.

The Weibull modulus values for the APC brackets using the conventional primer are lower than in previous studies (Bearn *et al.*, 1995; Willems *et al.*, 1997), suggesting a larger spread in the distribution of bond strength results. However, the characteristic strength is considerably higher. The clinical implications of these results are confusing. As we are really interested in the tail of the distribution it is helpful to investigate the bond strength at the 1 and 5 per cent chance of failure. A bond strength of 4 MPa for a 1 per cent chance of failure and 5·4 MPa for 5 per cent chance of failure are almost identical to values obtained in previous studies (Bearn *et al.*, 1995; Willems *et al.*, 1997). Expression of bond strengths at these percentage failure rates is a simple and clinically relevant means of describing the *in vitro* bond strength data, and would facilitate comparisons if used in future research. Quoting values for a 5 per cent chance of failure is preferable to a 1 per cent chance of failure, as statistically values at the very extremes of the Weibull distributions are likely to be less accurate. Ideally, these bond strengths should be expressed with confidence intervals.

This study could not demonstrate any relationship between the bond strength and the ARI category. The ARI describes the plane of failure after debonding. At one time it was felt that there was a relationship between the bond strength at the separate interfaces of the bonding system and the amount of residual debris left on the enamel surface. This presumed that an ARI value of 3, with all the adhesive left on the bracket was indicative of the weakest bond interface being between the adhesive and the bracket base. Whilst this is undoubtedly important it has been shown that the bracket base design and adhesive used must also play a part (O'Brien *et al*, 1988).

The results of this study suggest that bond strengths of brackets bonded using the hydrophilic primer are inferior to those bonded with the conventional primer. However, would the bond strength be adequate for clinical use? In a review of orthodontic bonding in 1975, Reynolds proposed that a maximum bond strength of 60–80 kg/cm^3 (6–8 MPa) would be required for successful clinical bonding, but that adhesives with an *in vitro* bond strength of approximately 50 kg/cm^3 (5 MPa) would be sufficient. According to Reynolds the median values of this hydrophilic primer would imply good clinical performance. However, we have discussed the importance of considering the whole spread of the data, particularly the weaker values (the tail of the distribution). The Weibull results for the hydrophilic primer are much less encouraging.

Conclusions

The bond strengths obtained with the insensitive primer were significantly lower than with the conventional primer. Although the median bond strength values were promising, the range of values obtained for this particular hydrophilic primer was disappointing when using the Weibull analysis. This analysis is thought to give a more realistic indication of the material's clinical performance. The real test is of course in the clinical situation, and a prospective randomized controlled clinical trial using this primer has recently been completed and will be reported in the future.

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