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Bonding to molars—the effect of etch time (an *in vitro* study)

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SUMMARY **This study evaluated the effect of etch time on the shear bond strength obtained when bonding to the buccal enamel of first molar teeth. Recently extracted first molar teeth were etched with 37 per cent phosphoric acid gel for 15, 30 and 60 seconds. Preformed cylinders of Concise composite resin were then bonded to the buccal surfaces of the molar teeth. After storage in water for 24 hours at 37°C, the specimens were debonded in a direction parallel to the buccal surface. Examination of the shear bond strengths showed significant differences in shear bond strength between 15 and 30 seconds ($P < 0.01$) and between 15 and 60 seconds ($P < 0.001$). Weibull analysis revealed that the probability of bond survival increased as etch time increased, however, the difference between 30 and 60 seconds was not statistically significant. The results indicate that, despite current recommendations of a 15-second etch for premolars, canines and anterior teeth, an etching time of at least 30 seconds should be used when bonding to the buccal surfaces of first molars. A further increase in etching time to 60 seconds produces no significant increase in bond strength.**

Introduction

Orthodontists routinely bond orthodontic brackets to the etched enamel of anterior teeth, canines and premolars. However, the acid etch technique (Buonocore, 1955) is less popular as a method of gaining attachment to molar teeth. Despite the periodontal health advantages of bonded molar attachments over molar bands (Boyd and Baumrind, 1992), their higher failure rate (Zachrisson, 1977; Gorelick, 1979) means that most clinicians prefer to place bands on molar teeth. The high failure rate of bonded molar attachments has usually been attributed to the larger masticatory forces posteriorly in the mouth and the difficulties in obtaining adequate isolation (Knoll *et al*., 1986).

The enamel structure present on the buccal surfaces of molar teeth may also be partly responsible for the reported higher failure rate of molar bonded attachments. Johnston *et al*. (1996) demonstrated that when an etch time of 15 seconds (which is commonly recommended for premolars and anterior teeth) was used on first molar teeth the etch patterns formed were of poor quality. An etch time of at least 30 seconds was required on first molars to produce an enamel micromorphology consistent with strong bonding. This longer etch time may be required to dissolve the prismless enamel present on the cervical two-thirds of molar teeth. This surface layer of prismless enamel is considered to be unsuitable for effective bonding (Arakawa *et al*., 1979; Gwinnett, 1981, 1992; McLaughlin, 1986).

The purpose of the present study was to determine whether varying the etch time influenced the shear bond strengths obtained when bonding to first molar teeth.

Materials and methods

Seventy-eight recently extracted first molar teeth were obtained from patients with a maximum age of 16 years. All the teeth included in the study had sound undamaged buccal surfaces. The teeth were stored in distilled water at room temperature before use. The roots of the teeth were mounted in a base of acrylic resin (Orthoresin, De Trey, Dentsply, Weybridge, UK) to facilitate positioning of the teeth in the debonding apparatus. To ensure that the debonding force was parallel to the mid-buccal surface, the teeth were mounted with the mid-buccal surface of the crown parallel to the long axis of the cylindrical acrylic base.

The buccal surfaces of the crowns were cleaned with a pumice and water slurry for 30 seconds followed by washing and drying. The area to be etched on each tooth was delineated by adhesive tape with a circular hole of 2.1 mm diameter. The adhesive tape was placed with the centre of the hole at the mid-buccal point of the crown where a molar bracket is usually placed. The tape was placed by a single experienced orthodontist (DJB).

The molar teeth were then randomly assigned into three groups of 26 teeth each and the acrylic bases were labelled with a unique identifying number. The exposed buccal enamel of each group was etched with 37 per cent phosphoric acid gel for 15, 30 or 60 seconds. The etchant was applied using the manufacturer's syringe tip and, after the appropriate time interval, each tooth was thoroughly washed for 30 seconds with water, then dried using an air syringe with an oil-free air supply.

The bonding procedure was carried out using preformed cylinders of composite resin (Concise, 3M Dental Products, St Paul, Minnesota, USA) of uniform dimensions (2.1 mm diameter of cylinder base and 6 mm height) formed using a tubular mould which ensured that the base surface areas of the cylinders were consistent. The cylinders of composite resin had been allowed to cure before being bonded to the exposed etched enamel using a fresh mix of Concise paste. Unfilled resin liner was not applied to the etched surface. The cylinders were placed on top of the molar buccal surface by a single operator (CDJ) using bracket tweezers. The molars were secured with their buccal surfaces facing upwards to obviate the need to support the composite cylinders during the curing process. Excess composite resin was removed with a sharp probe.

Following bonding of the composite cylinders to the molar buccal enamel, the composite bond was allowed to cure at room temperature for 15 minutes. The specimens were then stored in distilled water at 37°C for 24 hours before bond strength testing.

The tooth specimens were mounted by their acrylic bases in a mechanical testing machine (JJ Instruments 2000S, Lloyds Instruments PLC, Fareham, UK) and the shear bond strength of the composite specimens was tested using a cross-head speed of 1 mm/minute with the force applied parallel to the mid-buccal surface using a wire loop below the composite cylinder and immediately adjacent to the enamel. For each specimen the maximum load at failure was recorded and the enamel surface was examined using magnification to determine the site of bond failure.

Statistical analysis

The data obtained for load at failure were ranked, both within each group and for all observations, to establish whether there was a significant difference between each etch time and to carry out a Weibull analysis on the observed results. The shear bond strengths obtained were not normally distributed, but were well modelled by Weibull functions. A Weibull distribution may be used to predict the probability of survival of a composite cylinder at a given stress using the equation:

$$Ps = \exp - (\sigma - \sigma_u / \sigma_o)^{m} \qquad (1)$$

where Ps is the probability of survival, σ is the stress applied, σ_o is a constant, also known as the characteristic value, σ_u is the stress at which $Ps = 1$ and m is a constant called the Weibull Modulus (Ashby and Jones, 1988). The constant σ_u was taken as zero as in theory there is no lower limit of strength to which a defect within the composite cement may reduce a specimen.

If equation 1 is re-arranged and natural logarithms are taken of both sides twice, the equation is that of a straight line where the gradient is m (the Weibull Modulus) and the intercept with the *y* axis is equal to: m $\ln(\sigma_o)$.

The results of the shear bond strengths for each specimen were ranked and the experimental values of probability of survival (Ps_E) calculated according to the equation (McCabe and Carrick, 1986):

$$Ps_E = 1 - [n/(N + 1)] \qquad (2)$$

Hence, $Ps_E = (N + 1 - n)/(N + 1)$ where n = rank number of specimen and N = total number of specimens per group.

Natural logarithms were taken twice of the reciprocals of these probabilities of survival and also the natural logarithms of the stress at failure. A straight line was interpolated through the points and the correlation coefficients calculated, indicating the goodness of fit of the Weibull function for each experimental group. Using these values the functions predicting the probability of a composite cylinder surviving a given stress for each etch time were derived from the straight line gradients and y-axis intercepts.

The Kruskal–Wallis one-way analysis of variance by ranks test was also applied to these data. Comparison of the groups was then carried out using the Mann–Whitney test to determine significant differences between each pair of etch times.

Results

Examination of the teeth following debonding revealed that all failures occurred at the enamel-composite interface. The observed shear bond strengths are summarized in Table 1 and Figure 1.

Kruskal–Wallis analysis revealed a significant difference in the shear bond strengths obtained using the three different etch times ($P < 0.0005$), while Mann–Whitney tests showed significant differences in the shear bond strength between the 15-second etch group and the 30-second etch group ($P < 0.01$), and between the 15-second etch group and the 60-second etch group ($P < 0.001$).

Table 1 Observed failure loads.

Etch time (seconds)	Mean (MPa)	SD (MPa)	Range (MPa)
15	4.49	2.28	1.34–8.53
30	6.98	3.15	1.83–14.72
60	8.48	4.17	1.41–19.46

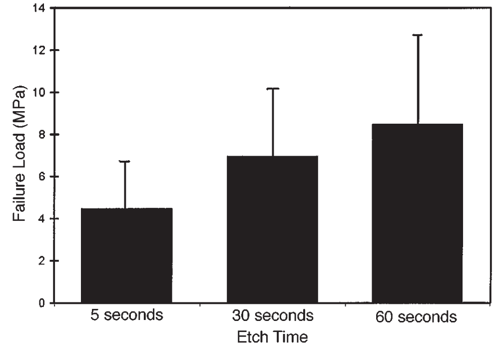

Figure 1 Mean and standard deviation of observed failure loads for 15-, 30-, and 60-second etch times.

However, the Mann–Whitney test failed to detect a significant difference in the shear bond strengths between the 30-second etch group and the 60-second etch group (Table 2).

Table 3 shows the results of the Weibull analysis as described above. Using these values, i.e. the Weibull modulus (m) and the characteristic value (σ_o), the functions predicting the probability of a cylinder surviving a given stress for each etch time were plotted in Figure 2. Thus, for any given applied stress the probability of survival for a composite cylinder bonded using each of the three etching times may be directly compared. Examination of Figure 2 reveals that the probability of survival increased between the 30- and 60-second etch, but the Mann–Whitney test indicated that this difference was not statistically significant.

The Weibull Modulus (m) gives an indication of the reliability of the shear bond strength for each etch time, with a low value indicating a wide distribution of results and a higher probability that a given specimen will have a low strength (Creyke *et al.*, 1982). Thus, the ranking for each

Table 2 Mann–Whitney tests of failure loads.

Etch time (seconds)	15	30	60
15	–	$P < 0.01$	$P < 0.001$
30	–	–	NS

NS, not significant.

Table 3 Weibull analysis of failure loads.

Etch time (seconds)	Number of specimens	Weibull modulus (m)	Characteristic value (σ_o)	Correlation coefficient
15	26	1.84	5.15	0.951
30	26	2.14	7.97	0.989
60	26	1.81	9.76	0.974

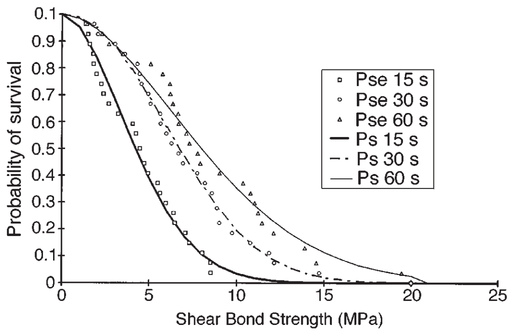

Figure 2 Probability of survival versus bond strength. *Ps* = predicted values. Ps_E = experimental values.

etch time for this parameter from best to worst was 30, 15, and 60 seconds.

Discussion

Bond failure of orthodontic attachments usually occurs at the bracket-resin interface or at the resin-enamel interface (Wang and Lu, 1991; Wang *et al.*, 1994). The present investigation aimed to evaluate the bond strengths between composite resin and molar buccal enamel. Therefore, preformed composite cylinders rather than orthodontic brackets were employed to avoid the possible confounding influence of bracket-resin failure on the results. Although the surface area of each cylinder base was less than that of an orthodontic bracket, this did not affect the results which were measured in units of force per unit area. In the present study all the bond failures occurred at the enamel-composite interface. Also, the enamel surface was not modified prior to etching other than the normal clinical practice of cleaning with pumice. Other studies have used ground enamel surfaces to produce a more standardized enamel surface for bonding (Legler *et al.*, 1989; Britton *et al.*, 1990; Holtan *et al.*, 1995). However, such a procedure is not representative of clinical practice (Fox *et al.*, 1994).

The general consensus from numerous studies of orthodontic bonding is that a 15 second etch time is adequate for orthodontic purposes (Barkmeier *et al.*, 1985; Wang and Lu, 1991; Wang *et al.*, 1994). However, these previous studies based on non-molar teeth have assumed that molar teeth respond similarly to acid etching. SEM analysis of surface micromorphology has shown that an etch time of 30 seconds is required to produce a satisfactory etch pattern on molar teeth (Johnston *et al.*, 1996).

The results of the present study indicate that increasing the etching time from 15 to 30 seconds significantly increased the probability of bond survival of the adhesive interface between the resin and molar buccal enamel. A further increase of etching time to 60 seconds increased the probability of bond survival, but not to a statistically significant extent. A similar trend has been noted in an earlier study of enamel micromorphology by Johnston *et al.* (1996). This previous study found a significant difference in etch quality between 15 and 30 seconds, but no significant increase between 30 and 60 seconds.

It has been shown that an etching time greater than 30 seconds used on anterior teeth may result in an increased likelihood of enamel fragment detachment during debonding (Wang and Lu, 1991). It has also been demonstrated that a double exposure of premolar enamel to phosphoric acid may actually reduce the bond strength (Gerbo *et al.*, 1992). It is therefore possible that an excessive etching time may reduce the strength of the etched enamel surface resulting in detachment of enamel fragments with a reduction in bonding strength. In the current study, the observed bond strengths do not indicate a reduction in bond strength at 60 seconds. However, the reduction in the Weibull Modulus from 2.14 at 30 seconds to 1.81 at 60 seconds indicates that the adhesive bond becomes less reliable with the increase in etching time producing a wider range of shear bond strengths. This may reflect the alteration in enamel micromorphology with prolonged etching, leading to a less predictable adhesive interface. The 30-second etch time produced a more consistent bond strength on the buccal surface of first molars.

Conclusions

A 15-second etch time is inadequate for achieving the best bond strength on molars, despite being widely recommended for premolar and anterior teeth. The results of the present study indicate that when using 37 per cent phosphoric acid gel, an etching time of at least 30 seconds should be used clinically when bonding to the buccal surfaces of first molars. Increasing the etching time from 30 to 60 seconds did not significantly improve the bond strengths obtained.

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