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Laboratory evaluation of a self-etching primer for orthodontic bonding

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SUMMARY The aim of the present study was to compare the mean bonding time, mean shear bond strength and mean survival time of stainless steel brackets with a micro-etched base bonded with a light-cure composite using a self-etching primer (SEP) or a conventional two-stage etch and prime system.

Brackets were bonded to 30 premolars with each bonding system. The bonding time was recorded for each specimen using a stopwatch. After storage in a humidor at 37°C for 24 hours, the shear debonding force was measured at a crosshead speed of 0.5 mm/minute. Another 10 premolars were bonded with each bonding system and used to assess survival time following the application of mechanical stress in a ball mill for 100 hours.

The mean bonding time of the SEP group (111.5 seconds) was significantly less than that of the two-stage bonding group (170.5 seconds) [mean difference 59 seconds; 95 per cent confidence interval (CI) 51.8–66.2 seconds, two sample t-test $P < 0.001$]. The mean shear bond strength of the SEP group (2.88 MPa) was significantly less than that of the two-stage bonding group (3.71 MPa) (mean difference 0.83 MPa; 95 per cent CI 0.23–1.42 MPa; two sample t-test $P = 0.008$). For the survival study, only one of the two-stage bonding group failed within 1 hour in the ball mill. The SEP significantly reduced bracket bonding time. The mean shear bond strength of the brackets bonded with the SEP was significantly less than those bonded with a conventional two-stage etch and prime system. There was no difference in survival time of brackets bonded by each bonding system.

Introduction

The introduction of acid etching of enamel by Buonocore (1955) afforded the possibility of direct bonding of orthodontic brackets to enamel as part of routine fixed appliance therapy (Newman, 1965). Nowadays, chemical- and light-cured adhesive resins are used universally for routine bonding of fixed appliances (Mandall *et al*., 2002) using conventional two-stage enamel etching and priming.

The continuing developments in dental material science have led to improvements in adhesive bonding formulations, resulting in the current availability of a wide range of products, including the single-step etch/primer solutions. These bonding systems combine an etchant conditioner and a primer resin agent for simultaneous use (Nishida *et al*., 1993). The main feature of the single-step etch/primer bonding systems is that no separate acid etching of the enamel is required; the liquid adhesive agent itself has an acid component that demineralizes the tooth structure in the same manner as 30–50 per cent phosphoric acid used in a conventional acid-etching technique (Miller, 2001). The combination of etching and priming steps into a single procedure means fewer stages in the bonding process, resulting in time saving for the clinician which has cost implications (Bishara *et al.*, 2001).

One of the newly introduced single-step adhesive bonding products is the self-etching primer (SEP) (Brosnihan and Safranek, 2000). Originally developed as the Prompt L Pop adhesive system (ESPE America Inc., Plymouth, PA, USA), it has been modified and is now marketed by 3M Unitek (Monrovia, CA, USA). The SEP can only be used with light-cured composites (Bond and Croll, 2001). Since its introduction, the SEP has been used in many dental applications (Croll, 2000).

It would appear, however, that bracket bonding with the SEP has been compared with a conventional two-stage bonding system in only one laboratory investigation (Bishara *et al*., 2001). Brackets bonded with the SEP were found to have a significantly lower mean shear bond strength compared with those bonded with a conventional two-stage adhesive system. The study, however, did not compare bonding time for each adhesive system. In addition, no reports of randomized clinical trials exist comparing the SEP with a conventional two-stage bonding system. Laboratory studies should, therefore, subject specimens bonded with the SEP to simulated mechanical stress in an attempt to predict their likely clinical performance and provide comparative data with conventional two-stage bonding systems.

The aim of the present study was to compare the mean bonding time, the mean shear bond strength and the mean survival time of stainless steel brackets with micro-etched base bonded with a light-cured composite (Transbond XT, 3M Unitek) using SEP or a conventional two-stage etch and prime system.

Materials and methods

Eighty premolars were collected which had been freshly extracted for orthodontic purposes. All teeth were removed from adolescent subjects, resident in an area with a non-fluoridated water supply. Subject age and background fluoride in the water supply will influence surface enamel characteristics and therefore the likely etch pattern obtained for bonding. The selection criteria adopted minimized bias from these variables. Each tooth was free of any enamel hypoplasia or restoration and had intact, sound buccal enamel. Following extraction, the teeth were decontaminated in 0.5 per cent Chloramine-T for 1 week before being transferred to storage in distilled water at 4ºC for up to 5 months as per the recommendation of the International Standards Organisation (1991).

For the bonding time and bond strength testing study, two groups were formed:

Group 1: 30 teeth (15 maxillary and 15 mandibular premolars) were bonded using the SEP.
Group 2: 30 teeth (15 maxillary and 15 mandibular premolars) were bonded with the conventional two-stage etch and prime system.

For the survival time study, another two groups were formed:

Group 1: 10 teeth (five maxillary and five mandibular premolars) were bonded using the SEP.
Group 2: 10 teeth (five maxillary and five mandibular premolars) were bonded with the conventional two-stage etch and prime system.

Bonding time and bond strength study

All bonding procedures were undertaken by one operator.

The root of each extracted premolar was grooved in the apical third with a diamond bur and then mounted to below the amelocementum junction in a block of self-curing acrylic, with the long axis vertical. After setting, the buccal surface of each tooth was cleaned using a fluoride-free pumice slurry, then washed with distilled water and dried in a stream of oil-free compressed air. A 0.022 inch slot stainless steel pre-adjusted edgewise premolar bracket with micro-etched base (3M Unitek) was bonded to the mid-buccal surface of each tooth. The brackets were kept in the manufacturer's packaging until immediately prior to bonding and were handled at all times with bonding tweezers to avoid contamination of the bonding base.

A conventional light-cured resin (Transbond XT) was used for bracket bonding. The resin adhesive was applied to the bracket base following tooth conditioning with either the SEP or a conventional two-stage etch and prime system (Transbond XT). The unit dose set-up of the SEP is designed for bonding an entire dental arch, although a single package can be used for both arches. This new single use adhesive system incorporates a pair of discrete reservoirs in a single sheath device combined with a disposable applicator tip and compartment. The first reservoir contains methacrylated phosphoric acid derivatives (esters), photosensitizers and stabilizers; the second reservoir contains water and soluble fluoride components. The third compartment houses an applicator tip. The advantage of this delivery system is that the separation of the reservoir pockets ensures that there is no possible mixing of the components before application, thus extending the product's shelf-life (Issa *et al*., 2000). The system is activated by successive operations of squeezing and folding to eject the solution of the first reservoir to the second reservoir. The liquid mixture is then ejected to a compartment that houses the applicator tip. The adhesive is then applied via the applicator microbrush by rubbing the enamel with rotating movements for 3–5 seconds. A gentle stream of air is then applied, facilitating solvent evaporation and ensuring that a glossy surface appearance is produced. The adhesive liquid mixture remains active for 1 hour.

Group 1. Thirty brackets were bonded using the SEP. The buccal surface of each tooth was etched/primed in a single stage by rubbing the enamel with the microbrush applicator for 5 seconds, followed by drying lightly using oil-free compressed air as recommended by the manufacturers. Composite resin (Transbond XT) was then applied to the bracket base and the bracket positioned firmly on the tooth surface. Excess composite was then removed from around the bracket base with a sharp dental probe prior to curing with an Ortholux light unit (3M Unitek) for 40 seconds (20 seconds from the mesial and 20 seconds from the distal aspect of each bracket). The same Ortholux conventional light-curing unit was used to bond all 80 teeth in this study.

Group 2. Thirty brackets were bonded with the conventional two-stage etch and prime system. The mid-buccal enamel of each tooth was etched for 15 seconds with 37 per cent orthophosphoric acid gel applied with a sponge pledget. Following rinsing with distilled water and drying (until the enamel appeared 'frosty') with oil-free compressed air, Transbond XT primer was applied to the etched surface and light cured for 10 seconds using an Ortholux light unit. Transbond XT composite was then applied to the bracket base, the bracket placed firmly in position and excess composite removed prior to light curing as per group 1. All specimens were then immersed in distilled water, each group in a separate container, and placed in a humidor at 37ºC for 24 hours prior to bond strength testing.

The time to bond each bracket was recorded using a digital timer (Whatman International Ltd, Maidstone, UK). The time in seconds was recorded from the application of the SEP or the orthophosphoric acid etching gel until the bonding composite had been cured.

The shear vertical debonding force was then measured for each specimen using a Nene M3000 Universal Testing machine (Nene Instruments Ltd, Wellingborough, UK) with a crosshead speed of 0.5 mm/minute. Each specimen was removed from the distilled water container (of the humidor) and mounted immediately in the testing apparatus. A steel loop, which fully engaged the gingival tie wing slot of the bracket, was connected to the crosshead of the Nene machine. Debonding took place according to the method of Fox *et al.* (1991). For each specimen, the shear debonding force value (N) was then divided by the bracket surface area (20.618 mm^2) to allow the calculation of bond strength (MPa). The surface area was supplied by the bracket manufacturer.

Survival time study

For the survival time study, each group of teeth was bonded using either of the adhesive systems as described earlier. Following the bracket bonding procedure, the specimens were immersed in distilled water and placed in a humidor at 37ºC for 24 hours before being subjected to mechanical stress in a ball mill. For survival time testing, specimens were placed in a 500 ml capacity cylindrical ceramic ball mill which contained 470 g of steatite spheres and 250 ml of distilled water at 37°C. The ball charge comprised 30 balls—23 had diameters in the range of 16–18 mm; two had diameters between 24 and 26 mm and five had diameters between 26 and 28 mm. The ball mill operated at 100 rpm for 100 hours.

After each hour of testing, the failed specimens (debonded brackets) were removed from the mill. The distilled water was replaced with a fresh sample at 37°C and testing recommenced and continued for 100 hours.

Statistical analyses

The mean bonding time and mean bond strength values for the two groups were compared using a two sample *t*-test. Weibull analysis (Weibull, 1951) was used to calculate the probability of failure at given values of applied force. The use of Weibull analysis takes account of the bond strength values at the extremes of the distribution and is used to calculate the probability of failure at given values of applied force. A Weibull modulus value can then be generated for each specimen group, allowing numerical evaluation of the 'dependability' of each bonding system.

Results

Bonding time

The mean bonding time for the SEP group was 111.5 seconds (range 84–140 seconds) while that for the two-stage group was 170.5 seconds (range 142–205 seconds). The mean difference between the two groups was 59 seconds [95 per cent confidence interval (CI) 51.8–66.2 seconds].

The bonding time of the SEP group was significantly less than that of the conventional two-stage etch and prime group ($P < 0.001$).

Shear bond strength

The mean shear bond strength for the SEP group was 2.88 MPa (range 1.41–4.08 MPa) while that for the two-stage group was 3.71 MPa (range 1.76–7.43 MPa). The mean difference between the two groups was 0.83 MPa (95 per cent CI 0.23–1.42 MPa).

The mean shear bond strength of the SEP group was significantly less than that of the conventional two-stage etch and prime group ($P = 0.008$).

Weibull analysis (Figure 1)

A higher Weibull modulus ($m = 4.00$) was recorded with the conventional two-stage bonding system, indicating greater bond reliability with this bonding system compared with the SEP ($m = 3.50$).

The Weibull curves indicate that for a given probability of failure, significantly less force would be required to dislodge a bracket bonded with the SEP ($R^2 = 0.99$) compared with one bonded with the conventional two-stage system ($R^2 = 0.98$).

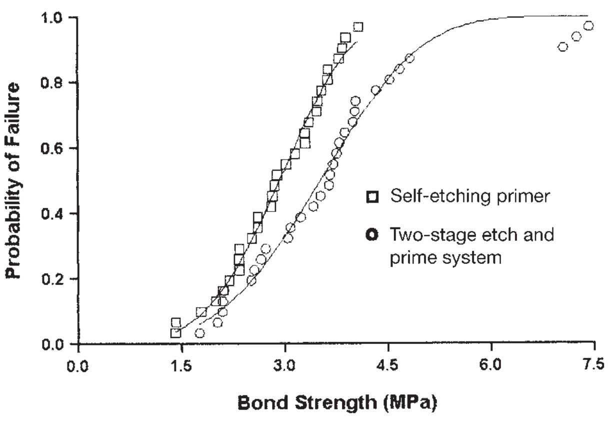

Figure 1 Weibull curves for brackets bonded with self-etching primer ($m = 3.50$; $R^2 = 0.99$) and the conventional two-stage etch and prime system ($m = 4.00$; $R^2 = 0.98$).

Survival time

Only one bracket bonded with the conventional two-stage system failed within 1 hour in the ball mill. No other bond failures were recorded, indicating no difference between the two adhesive systems.

Discussion

Tooth storage, preparation and sample size per adhesive group used in the bond strength studies followed guidelines in the orthodontic literature (Fox *et al.*, 1991; International Standards Organisation, 1991; Eliades and Brantley, 2000). One operator (YA) conducted all bonding procedures to standardize this variable.

The mean bonding time for brackets bonded with the SEP was almost one-third less than that of brackets bonded with the conventional two-stage etch and prime system; this was statistically significant. Bonding brackets with the SEP is likely, therefore, to reduce clinical chairside time for both clinicians and patients, which will help to improve overall clinical cost-effectiveness.

Full-size stainless steel (0.022 inch slot) pre-adjusted edgewise premolar brackets with a micro-etched base were used in this study as micro-etching has been shown to improve bond strength (Millett *et al.*, 1993). Bond strength testing in this investigation was performed according to Fox *et al.* (1991). Testing at 24 hours was chosen as it has been widely reported previously, and permits comparison with other *ex vivo* bond strength studies. The gingivo-occlusal direction of the shear debonding force used is similar to that in other research studies (Millett *et al.*, 1999; Sargison *et al.*, 1999; Linklater and Gordon, 2001). Application of a different direction of debond force (occluso-gingival) has, however, been employed in other investigations (Bishara *et al.*, 1999, 2001; Graf *et al.*, 1999).

The mean shear bond strength for brackets bonded with the SEP was almost one-quarter less than that of brackets bonded with the conventional two-stage etch and prime system; this was statistically significant. This confirms the findings of Bishara *et al.* (2001). That study, however, used extracted human molars rather than premolars. Other important differences between the present investigation and that of Bishara *et al.* (2001) include different regimes for tooth disinfection and storage, different tooth etching/priming times for bonding with the SEP, a different direction of bracket shear debonding force application, as well as different crosshead speeds.

The aetiology of the decreased bracket bond strength for the SEP compared with that of the conventional two-stage bonding system is unknown. Possible reasons may be the difference in chemical composition and concentration of the etchant between the two systems. The SEP uses phosphoric acid esters whose concentration is not given in the marketed product literature, whereas the conventional two-stage bonding system is based on 37 per cent orthophosphoric acid. In addition, the mode of etching/priming between the two bonding systems is also different (simultaneous etching/priming with the SEP versus separate etching and priming stages for the conventional two-stage bonding system).

A Weibull analysis was used to calculate the probability of failure at given values of applied force. This indicated that for a given probability of failure, significantly less force would be required to dislodge a bracket bonded with the SEP compared with one bonded with the conventional two-stage bonding system. A higher Weibull modulus was recorded for brackets bonded with the conventional two-stage bonding system, indicating greater bond reliability with this bonding system than with the SEP.

Although there was a statistically significant difference in mean shear bond strength between the two adhesive systems, only one bracket bonded with the conventional two-stage system failed (within 1 hour) in the ball mill. This bond failure could be the result of a single impact event. With no further bond failures recorded in the ball mill, it is equally possible that the force applied in milling was below any threshold for fatigue.

The different methods of bond testing assessment used in this investigation produced different results. A previous study (Durning *et al.*, 1994) has shown a similar dichotomy between bond strength testing and survival time testing using the ball mill. This technique has proven useful in predicting the clinical performance of some orthodontic materials (Millett *et al.*, 1993; Abu Kasim *et al.*, 1996). The ball mill employs diverse forces of varying magnitude (Tarasiewicz and Radzisewski, 1989a,b) with bond failure probably occurring through a process of slow crack propagation generated within the bonding material by the force of impact and mechanical action of the ceramic spheres (Abu Kasim *et al.*, 1996).

Further work is required to assess if the performance of the two bracket bonding systems evaluated in this laboratory study is mirrored in the clinical environment.

Conclusions

The mean bonding time and the mean shear bond strength of brackets bonded with the SEP were significantly less than those of the two-stage bonding group. The mean survival time of brackets bonded with the SEP or the conventional two-stage bonding system were similar.

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