Shear bond strength of fluoride-releasing orthodontic bonding and composite materials

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SUMMARY Several fluoride-releasing bonding materials are available for orthodontic bracket placement. These are supposed to prevent white spot lesions during therapy. The objectives of this *in vitro* study were to evaluate the shear bond strength (SBS) and failure mode of a recently introduced fluoride-releasing adhesive, as well as the comparison with established orthodontic adhesives. Sixty bovine mandibular incisors were randomly allocated to three groups (n = 20): stainless steel brackets were bonded with TransbondTM Plus Color Change Adhesive, TransbondTM XT, or Light BondTM. A universal testing machine was used to determine the SBS at a crosshead speed of 1 mm/minute. After debonding, the adhesive remnant index (ARI) was used to assess the adhesive remaining on the brackets.

One-way analysis of variance comparing the three experimental groups showed no differences between the bonding systems for mean SBS (P = 0.27). ARI scores showed more residual adhesive on the teeth bonded with the TransbondTM systems (P < 0.01). As the fluoride-releasing bonding system provided sufficient mean bond strength *in vitro* (19.9 MPa), it may be used as an additional prophylactic measure in orthodontic therapy. However, the clinical effectiveness of its fluoride release may be questionable, as the amount of fluoride required from a bonding material to be caries preventive is still unknown.

Introduction

Stable attachments are a prerequisite for successful orthodontic therapy. Since fixed appliances facilitate the retention of bacterial plaque (Sukontapatipark et al., 2001) and hamper oral hygiene (Naranjo et al., 2006), avoiding dental decay during treatment is a key issue in orthodontics (Sonis and Snell, 1989). In spite of advanced materials and treatment devices, fixed appliances still carry an elevated risk of distinctive white spot lesions adjacent to brackets (Sukontapatipark et al., 2001). Their prevalence has been reported to be as high as 97 per cent (Boersma et al., 2005). These lesions are precursors of enamel caries caused by demineralization of the tooth by organic acids produced by cariogenic bacteria (Featherstone, 2004). Hence, an ideal adhesive system should have sufficient bond strength to withstand untimely impact forces on bonded brackets and, at the same time, prevent decalcification. Based on the analysis of the cariostatic mechanisms of systemic and topical fluorides, the development of clinical procedures to establish and maintain low levels of free fluoride in the oral cavity for preventing dental decay has been suggested (Margolis and Moreno, 1990; Marinho et al., 2003). Especially in orthodontic patients, fluoride delivery may be beneficial (Benson et al., 2004). Various methods of administering fluoride during orthodontic treatment have been used, including toothpastes, mouth rinses, gels, and varnishes. In addition, materials have been introduced delivering fluoride during treatment such as fluoridereleasing composite bonding materials (resin modified),

glass ionomer cements (GIC), compomers, slow-release fluoride devices, and fluoride-releasing elastomeric ligatures (Benson *et al.*, 2004).

Recently, TransbondTM Plus Color Change Adhesive (3M Unitek) was introduced. The adhesive contains a fluorosilicate glass as the fluoride source. The hydrophilic nature of the adhesive allows fluoride diffusion through the cured cross-linked matrix in an aqueous medium (Tzou and Darrell, 2007). Its pink colour may provide a visual aid for bracket positioning and excess removal of the adhesive. Upon light curing, the colour immediately fades. No original scientific data are available to date on the bond strength of this adhesive system.

The objectives of this *in vitro* study were to evaluate the shear bond strength (SBS) and failure mode of Transbond[™] Plus Color Change Adhesive and to compare the results with those of two established orthodontic adhesives (Table 1). The null hypotheses were as follows:

- 1. There is no difference in SBS between the three tested adhesives.
- 2. There is no difference in the failure mode between the three groups in terms of the adhesive remnant index (ARI; Årtun and Bergland, 1984).

As the two tested fluoride-releasing adhesives were applied after different enamel preparations [conventional etching versus self-etching primer (SEP)], any influence of these procedures on SBS and failure mode was also evaluated.

Materials and methods

Sixty permanent incisors were extracted from lower bovine jaws obtained from a local abattoir. After manual removal of adherent soft tissue using a disposable scalpel, the teeth were stored in a disinfectant solution of 0.5 per cent chloramine-T at 6°C. The storage time varied from 7 to 9 weeks. The tooth surfaces were inspected for any gross evidence of fracture or caries. All teeth were cleansed with a brush and oil-free pumice at slow speed for 10 seconds and rinsed with tap water before random assignment to three groups of 20 specimens each. Sixty stainless steel maxillary lateral incisor brackets (0.018 inch Mini Diamonds, Ormco, Orange, California, USA) were used in this study. The area of the bracket base was 10.56 mm², ascertained by measuring the dimensions of six brackets.

Both TransbondTM Plus Color Change Adhesive and TransbondTM XT (3M Unitek) were used in combination with TransbondTM Plus SEP (3M Unitek). Following the removal of excess water on the tooth surface, the SEP was activated according to the product description and applied for 10 seconds. An air burst of 2 seconds was delivered using an oil- and moisture-free air source to achieve a thin film of the primer liquid. After applying one of the two adhesives, the paste was gently pressed onto the bracket base with a spatula. The bracket was then immediately placed onto the tooth surface.

The Light Bond[™] group was treated with Gel Etching Agent and a sealant resin (Reliance Orthodontic Products). Subsequent to thorough air drying of the tooth surface, 37 per cent phosphoric acid was applied for 30 seconds. The tooth was rinsed with water for another 30 seconds and dried until the etched enamel appeared chalky white. A thin uniform coating of sealant resin was applied with a disposable brush, gently air-dried, and polymerized with an Ortholux light emitting diode curing light (3M Unitek) for 30 seconds. The application of the adhesive paste as well as the placement of the bracket was the same as for groups 1 and 2. The protocols are listed in Table 2.

When placing the brackets, a force of 300 g was applied for 5 seconds using a spring balance (Correx Tension Gauge, Haag-Streit AG, Koeniz, Switzerland) and ensuring a uniform thickness of the adhesive (Eliades and Brantley, 2000). Excess adhesive was removed with a scaler. The adhesive was light cured for 40 seconds (10 seconds each from the mesial, distal, cervical, and incisal). The efficiency of the lamp was tested prior to each curing cycle using the meter within the unit. A 0.018 \times 0.025 inch stainless steel wire (Ormco) was laser welded to each bracket slot to minimize bracket deformation during the testing procedure (Klocke and Kahl-Nieke, 2006). The prepared specimens were then stored in water at 37°C. The bonded teeth were embedded in type 3 dental stone (Moldano blue; Heraeus

Table 1	Description	of the bonding	materials used.

Group	Test material	Туре	Batch number	Manufacturer
1	Transbond [™] Plus Self-Etching Primer Transbond [™] Plus Color Change Adhesive (capsules)	Fluoride-releasing self-etching primer Fluoride-releasing resin-based composite adhesive	291 800C AG	3M Unitek Orthodontic Products, Monrovia, California. USA
2	Transbond™ Plus Self-Etching Primer Transbond™ XT Light Cure Adhesive (capsules)	Fluoride-releasing self-etching primer Resin-based composite adhesive	291 800C YA	
3	Gel Etching Agent Light Bond [™] Sealant Resin Light Bond [™] Adhesive Paste (capsules)	37% phosphoric acid Fluoride-releasing sealant resin Fluoride-releasing resin-based composite adhesive	0703277 0600609 0704754	Reliance Orthodontic Products, Itasca, Illinois, USA

 Table 2
 Bonding protocols according to the manufacturers' instructions.

	Transbond [™] Plus Color Change Adhesive	Transbond [™] XT	Light Bond TM
Polishing	10 seconds	10 seconds	10 seconds
Enamel etching	_	-	30 seconds
Rinsing	_	-	30 seconds
Air drying	-	-	Until enamel is chalky white
Application of sealer	-	-	+
Etching and priming in one step	10 seconds	10 seconds	_
Air drying	2 seconds	2 seconds	+
Light curing	_	-	30 seconds
Application of composite and positioning of bracket	+	+	+
Light curing	40 seconds	40 seconds	40 seconds

Kulzer, Hanau, Germany) with the bracket bases aligned perpendicular to ensure a load application parallel to the bracket base. An incisal-to-cervical shear force was applied as close to the bracket–tooth interface as possible (Figure 1) by a chisel-shaped rod attached to the crosshead of a universal testing machine (Zwick, Ulm, Germany). Testing was performed approximately 3 hours after the preliminary bonding procedure at a crosshead speed of 1 mm/minute (Jonke *et al.*, 2008). The load at failure was recorded with the testXpert V11.0-software (Zwick) and calculated in megapascals (MPa) by dividing the shear force (Newton) by the area of the bracket base (square millimetres).

After debonding, the failure surfaces were examined using an optical stereomicroscope (Vision Engineering Ltd, Woking, Surrey, England) at a magnification of ×10. The mode of failure was assessed using the ARI (Årtun and Bergland, 1984), which defines the mode of bond failure between the enamel, adhesive, and bracket base (Table 3). Bonding, shear testing as well as ARI scoring were assessed by the same operator (BCP). The resulting bond strengths of the three groups were compared by a one-way analysis of variance (ANOVA). A chi-squared test was used to determine significant differences in the ordinal ARI scores. All statistical tests were run with a predetermined significance level of $\alpha = 0.05$.

Results

The results of the ANOVA comparing the SBS of orthodontic brackets to bovine teeth with the three adhesive systems are given in Table 3. There was little difference between the mean and median bond strengths values. There was no evidence suggesting a statistical difference in the mean SBS between the groups (F = 1.35; P = 0.27).

The distribution and results of the chi-squared analysis of the ARI scores are illustrated in Table 4. The chi-squared test showed a highly significant difference between ARI scores of the TransbondTM Plus and Light BondTM groups. Fifty per cent of the enamel surfaces in the Light BondTM group showed less than half of the adhesive remaining after debonding, indicating more failure at the enamel–adhesive interface. The TransbondTM Plus group showed more than half of the adhesive remaining in 95 per cent, indicating failure at the adhesive–bracket interface. Enamel fractures were not observed in any of the three groups.

Discussion

The null hypothesis that there is no difference in bond strength between the groups was accepted. The new fluoridereleasing TransbondTM Plus Color Change Adhesive provided the same high bond strength as TransbondTM XT. The latter has been used regularly as a control (Scougall Vilchis *et al.*, 2007). In the present study, both were used in



Figure 1 Experimental setting: a chisel-shaped rod applied the shear force as close to the bracket-tooth interface as possible. The bovine teeth were embedded in dental stone.

 Table 3
 Results of one-way analysis of variance comparing shear bond strengths between the groups.

Adhesive	Shear bond strength (MPa)			
	Mean ± SD	Range	Median	
Transbond [™] Plus Color Change	19.9 ± 4.3	10.0–24.1	21.7	20
Transbond [™] XT Light Bond [™]	21.6 ± 5.3 22.1 ± 3.9	9.5–29.7 9.5–26.8	22.0 23.3	20 20

Groups were not statistically different from each other (F = 1.35; P = 0.27).

combination with a fluoride-releasing SEP. Bond strengths of Transbond[™] Plus Color Change Adhesive were also comparable with those obtained with the fluoride-releasing Light Bond[™] adhesive, which was applied in combination with phosphoric acid and a sealant resin. With the introduction of this third well-established orthodontic adhesive, it was possible to assess the performance of a SEP versus conventional enamel conditioning. The acid etch

Table 4 Distribution and results of chi-squared test of adhesive remnant index (ARI) scores ($\chi^2 = 12.83$; P < 0.05).

Adhesive	ARI				
	0	1	2	3	
Transbond [™] Plus Color Change	—	1	12	7	20*
Transbond [™] XT	2	3	11	4	20
Light Bond [™]	3	7	9	1	20*

0: no adhesive remaining on the tooth, failure between adhesive and enamel; 1: less than half of the adhesive left on the tooth; 2: more than half of the adhesive remaining on the tooth; 3: all adhesive left on the tooth with distinct impressions of the bracket mesh; failure between adhesive and bracket base.

*Groups were statistically different from each other (P = 0.0034).

regimen is widely presumed to produce the optimal bond of composite resin to enamel (House et al., 2006). In fact, this system also showed the highest mean bond strength in this study, a finding, however, which was not statistically significant. Still, the number of steps required with this bonding process and the moisture sensitivity of the technique identified the need for the development of SEPs (House et al., 2006). Originally designed for restorative dentistry, Transbond[™] Plus SEP proved to be reliable and compatible with both adhesives tested in the setting of this study. Numerous in vitro (Hirani and Sherriff, 2006; Ritter et al., 2006; Vicente and Bravo, 2006; Vicente et al., 2006; Faltermeier et al., 2007; Turk et al., 2007) and in vivo (Cal-Neto et al., 2006; Dos Santos et al., 2006; Manning et al., 2006) studies have assessed the bond strengths or rather bond failures of this particular fluoride-releasing SEP versus the conventional etch and prime regimen and found the SEP to perform as well, if not better.

Bond strengths recorded in vivo are significantly lower than those achieved in vitro due to deterioration of the adhesive in the oral environment (Murray and Hobson, 2003). Moreover, essential factors such as stresses arising from an activated archwire coupled with occlusal loads, critical pH, and temperature variations cannot be replicated in a laboratory investigation (Eliades and Brantley, 2000). The fact that the load was applied as close to the brackettooth interface as possible resulted in high values in this study. For shear testing, a significant influence of the distance of force application from the enamel surface is evident (Thomas et al., 1999). The absence of thermocycling of the bonded teeth may also have contributed to these results. These facts confirm that inter-study comparisons of SBS are not appropriate because discrepancies in load location or debonding force angulation cannot be excluded (Klocke and Kahl-Nieke, 2006). However, several bond strength studies (D'Attilio et al., 2005; Korbmacher et al., 2006; Scougall Vilchis et al., 2007) based their conclusions on comparisons with values suggested to be adequate for clinical use by previous investigators. Although they are frequently cited, these proposed stress values are not evidence based (Thind *et al.*, 2006). Inconsistencies in tooth selection, storage conditions, enamel preparation, bonding, or testing were not taken into account (Eliades and Brantley, 2000). As no standardized and widely used bond strength assessment protocol exists, an intra-study comparison with

a control group seems to be a sensible approach. In spite of the similar bond strengths in this study, analysis of ARI scores showed a statistically significant disparity. Thus, the null hypothesis that there would be no difference in ARI scores between the groups was rejected. Although the measured bond strengths appeared to be high, no enamel fractures were detected. Within the SEP groups, the majority of ARI scores were 2 and 3, indicating failure primarily at the bracket-adhesive interface. Therefore, the adhesive bond to enamel and the cohesive strength of the adhesive were higher than the adhesive bond to the bracket base. Almost the converse was true for the conventional etch group, which differed significantly from the Transbond[™] Plus SEP combination. In vitro investigations of the locus of bond failure in comparisons of the performance of SEP and conventional etching have not produced a consensus view, i.e. whereas a large number of SEP studies showed bond failure to occur most frequently at the enamel-adhesive interface, others demonstrated that SEP produces a locus of bond failure similar to that of conventional etching (Thind et al., 2006). In this study, the application of adhesive resin following acid etching resulted in far less adhesive remaining on the enamel surface after in vitro debonding. This has been observed only in a few previous studies (Bishara et al., 2001; Thind et al., 2006). Less residual adhesive after debonding would be beneficial for the clean-up procedure at the end of treatment: it would save both time and prevent iatrogenic enamel loss (Årtun and Bergland, 1984). However, the locus of bond failure is determined by a complex combination of contributory factors including the direction of the force applied, enamel pre-treatment, the adhesive itself, and the bracket type (Katona, 1997).

Excess composite around the bracket base is the critical site for plaque accumulation due to its rough surface and the shrinkage gap at its periphery (Sukontapatipark et al., 2001). The initial pink colour of Transbond[™] Plus Color Change Adhesive may provide a visual aid for minimizing excess composite around the bracket base. The adhesive contains a dye that photobleaches when exposed to light. The incorporation of a fluoride source in the adhesive resin is intended to prevent white spot lesions. Bonding materials capable of preventing the undesirable effect of white spot formation, while maintaining adequate bond strength, would be an advance. Although white spots were shown to fade partially after removal of fixed appliances (Van der Veen et al., 2007), the overriding objective should be to prevent their development during orthodontic treatment. GIC would also satisfy the requirement of fluoride release (Benson *et al.*, 2004; Eliades, 2006), but as conventional GIC show disadvantages such as requiring mixing and relatively low fracture strength, they are of limited use in stressed areas (Glasspoole *et al.*, 2001a). On the other hand, there is evidence suggesting that resin-modified GIC may provide adequate bond strengths *in vitro* (Movahhed *et al.*, 2005) and clinically (Summers *et al.*, 2004).

Albeit the caries-preventive effects of fluoride are well known (Ten Cate and Featherstone, 1991), the exact amount of fluoride release from a dental material to be clinically effective is still unclear (Erickson and Glasspoole, 1995; Ten Cate, 2004). A single report (Tzou and Darrell, 2007) dealing with the fluoride release rate of TransbondTM Plus Color Change Adhesive indicated that the release rate dropped to half the initial level after 1 week and to one-third after 4 weeks. A statistically significant degree of enamel protection was found when comparing fluoride-releasing materials (GIC, fluoridereleasing composites) with non-fluoride control materials (Sonis and Snell, 1989; Glasspoole et al., 2001b). However, the data of two recent systematic reviews are controversial (Benson et al., 2004; Derks et al., 2004). While Benson et al. (2004) found evidence suggesting that a daily fluoride mouth rinse or fluoride-releasing bonding materials (especially GIC) reduced the severity of white spots, Derks et al. (2004) claimed that only the use of chlorhexidine or fluoride-containing toothpaste inhibited caries. Thus, the clinical use of a compliance-free fluoride-releasing bonding system may be regarded as an additional prophylactic measure in orthodontic therapy since its bond strength appears to be sufficient.

Laboratory testing based on recommendations in the literature (Eliades and Brantley, 2000; Klocke and Kahl-Nieke, 2006) is a necessity for the initial evaluation of bonding systems, although physical adhesive properties can only be clarified to a certain extent by *in vitro* approaches (Korbmacher *et al.*, 2006). Bovine enamel is a valid alternative to human enamel for SBS testing (Lopes *et al.*, 2003; Saleh and Taymour, 2003; Titley *et al.*, 2006; Krifka *et al.*, 2008), but the actual performance of a system has to be assessed in the environment for which it was intended (Eliades and Brantley, 2000).

Conclusions

- 1. The fluoride-releasing Transbond[™] Plus Color Change Adhesive performed as well as the two established adhesives, Transbond[™] XT and Light Bond[™], in terms of SBS.
- The fluoride-containing SEP used in combination with TransbondTM Plus Color Change Adhesive or TransbondTM XT showed sufficient bond strength compared with conventional etching combined with Light BondTM.
- 3. Comparison of the ARI scores indicated that there was significantly more residual adhesive remaining on teeth treated with the SEP.

4. These *in vitro* findings should be carefully extrapolated to the clinical setting.

Future clinical trials are needed for evidence-based recommendations on the optimal caries-preventive strategy since the amount of fluoride released from a bonding material to be clinically effective is still unknown.

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