The effect on shear bond strength of different antimicrobial agents after acid etching

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SUMMARY The aim of the present study was to determine whether the application of different primers containing antibacterial agents (Micro Prime[™], Seal&Protect[™], and Gluma Desensitizer[™]) can affect the shear bond strength (SBS) of an orthodontic resin composite.

Seventy-two crowns of extracted lower human incisors were mounted in acrylic resin leaving the buccal surface of the crowns parallel to the base of the moulds. The teeth were randomly distributed into three experimental and one control group, each containing 18 teeth. In each experimental group, the primers were applied to the etched enamel surfaces. In the control group, no antibacterial primer was used. An orthodontic composite resin was applied to the surface into cylindrical-shaped plastic matrices after application of an orthodontic adhesive primer (Transbond XT). For shear bond testing, a stubby-shaped force transducer apparatus (Ultradent) was applied at a crosshead speed of 1 mm/minute to each specimen at the interface between the tooth and the composite until failure occurred. A Kruskal–Wallis one-way ANOVA and a Mann–Whitney *U*-test with a Bonferroni adjustment were used for statistical analysis.

There was no significant difference between Seal&Protect[™] (27.98 ± 8.73 MPa) and the control (35.15 ± 7.85 MPa) (P > 0.05). However, Gluma[™] (21.61 ± 7.96 MPa) and Micro Prime[™] (14.89 ± 5.55 MPa) caused a decrease in bond strength (P < 0.05). No statistically significant difference was observed between Seal&Protect[™] and Gluma[™] (P > 0.05).

As triclosan containing Seal&Protect[™] did not cause a significant decrease in bond strength, it can potentially be used under an orthodontic resin composite to obtain an antibacterial effect. However, further *in vivo* studies are required.

Introduction

The components of an orthodontic appliance create new retention areas for micro-organisms and impede access to the tooth surfaces for optimal cleaning. Plaque formation is therefore more extensive in orthodontic patients undergoing treatment than in subjects without appliances, and high saliva bacteria concentrations have been reported (Bloom and Brown, 1964; Balenseifen and Madonia, 1970; Corbett *et al.*, 1981; Scheie *et al.*, 1984; Lundström and Krasse, 1987; Forsberg *et al.*, 1991, 1992). Gorelick *et al.* (1982) found that approximately 50 per cent of orthodontic patients experienced lesions on a tooth during treatment. Øgaard *et al.* (2001) indicated that the high prevalence of carious lesions may be due to the high cariogenic challenge prevailing in the plaque around orthodontic appliances.

Various antimicrobial agents have been incorporated into oral products and approved for intra-oral use. Most are designed to prevent plaque accumulation and thereby prevent or treat gingivitis. Glutaraldehyde, triclosan, and benzalkonium chloride remain the most effective antiplaque and anti-gingivitis agents (Geftic *et al.*, 1979; Gjermo, 1989; Moran *et al.*, 2000; Othman *et al.*, 2002).

To be accepted clinically, modified materials must provide superior antimicrobial activity and display comparable physical properties such as tensile and shear bond strength (SBS), when compared with conventional adhesives. Fluoride is the most common preventive additive in orthodontic adhesives. Conventional glass ionomer cements (GICs) have very low SBS and are not appropriate for routine orthodontic bonding (Ashcraft *et al.*, 1997). Recently, Jedrychowski *et al.* (1983), Ribeiro and Ericson (1991) and Imazato *et al.* (1994) modified filling materials by adding antimicrobial agents to composite resins, acrylic resins, and GICs. The authors found that these agents, added in minute amounts, could impart an antibacterial trait to dental materials without significantly affecting their physical properties. No orthodontic adhesives containing an antimicrobial agent are commercially available, despite the need for a material that combats the microbial attack on the adhesive and the tooth structure (Matasa, 1995).

Thorough plaque and inflammation control is very difficult in patients with fixed orthodontic appliances, and chemical agents in the form of mouth rinses or oral sprays have been shown to be useful adjuncts (Karaman and Uysal, 2004). Varnish forms of antibacterial solutions such as benzydamine, triclosan, and xylitol could be helpful in orthodontic patients for suppressing oral mutans or other microbe levels for a long period after their application when used before placement of fixed orthodontic appliances.

Therefore, the aim of the present study was to determine whether the application of different types of primer containing an antibacterial agent to the etched enamel surface will affect the SBS of orthodontic composite resins.

Materials and methods

Mandibular incisors extracted due to periodontal reasons were stored at +4°C in a physiological saline solution. Teeth with hypoplastic areas, cracks or gross irregularities of the enamel structure were excluded from the study. The criteria for tooth selection dictated no pre-treatment with a chemical agent such as alcohol, formalin, hydrogen peroxide, etc. Soft tissue remnants and calculus were removed from the teeth, following which they were cleaned with a fluoridefree pumice and rubber cup. Seventy-two extracted teeth were selected.

The roots of the teeth were cut off with a water-cooled diamond disk and the crowns mounted in a 3 cm diameter circle mould using chemically cured acrylic resin (Vertex, Zeist, The Netherlands). The crowns were mounted so that their labial surfaces were perpendicular to the base of the moulds. Prior to bonding, the labial surface of each tooth was polished for 1 minute with a combination of a polishing agent and a brush at a low speed (3000 r.p.m.) using a micro-motor.

The teeth were distributed into three experimental groups and one control group, each containing 18 teeth. A 37 per cent orthophosphoric acid gel (3M Dental Products, St. Paul, Minnesota, USA) was used for the acid etching of the teeth for 15 seconds. The teeth were then rinsed with water for 15 seconds and dried with oil-free air for 10 seconds until a frosty white appearance of the etched enamel was observed.

For each experimental group, one of three commercially available primers containing an antibacterial agent (Micro Prime[™], Seal&Protect[™], or Gluma Desensitizer[™]) was applied to the etched surfaces as shown in Table 1. In the control group, no antibacterial primer was used. An orthodontic adhesive primer (Transbond XT, 3M Unitek, Monrovia, California, USA) was used and light cured in all groups. An orthodontic composite resin (Transbond XT adhesive paste) was added to the surface by packing the material into cylindrical-shaped plastic matrices with an internal diameter of 2.34 mm and a height of 3 mm (Ultradent, South Jordan, USA) (Figure 1). Excess composite was carefully removed from the periphery of the matrix with an explorer. The composite was cured with a curing light (Hilux, Benlioğlu Dental, Ankara, Turkey) for 20 seconds. The intensity of the light was at least 400 mW/cm². The specimens (Figure 2) were then stored in distilled water at 37°C for 24 hours before bond strength testing. For SBS testing the specimens were mounted in a universal testing machine (Model 500, Testometric, Rochdale, Lancashire, UK) (Figure 3). A stubby-shaped force transducer apparatus (Ultradent) attached to a compression load cell and travelling at a crosshead speed of 1 mm/minute was applied to each specimen at the interface between the tooth and the composite until failure occurred. The notched blade was placed directly over the resin stub, flush against the tooth. The maximum load (N) was divided by the cross-sectional area of the bonded composite posts to determine SBS in MPa.

Fracture analysis

Fracture analyses were performed using an optical stereomicroscope (Olympus SZ4045 TRPT, Osaka, Japan). Failures were classified as cohesive if more than 80 per cent of the resin remained on the tooth surface, adhesive if less than 20 per cent of the resin remained on the tooth surface, or mixed if certain areas exhibited a cohesive fracture and other areas an adhesive fracture (Sengün *et al.*, 2002).

Statistical analysis

Descriptive statistics, including the mean, standard deviation, and minimum and maximum values, were calculated for each of the four groups of teeth. A Kruskal–Wallis one-way ANOVA and a Mann–Whitney *U*-test with a Bonferroni adjustment were used to analyse SBS differences between the groups at a significance level of $P \le 0.05$. Fracture modes were analysed using a Pearson chi-square test.

Results

The descriptive statistics for each group are presented in Table 2. The results of the Kruskal–Wallis ANOVA revealed

Table 1 Manufacturers' information, active ingredients, and application procedures of the antimicrobial agents used in the study.

Materials/manufacturers	Active ingredients	Procedures
Micro Prime™ (Danville Engineering, California, USA; lot 1424)	Benzelkonium chloride and HEMA	a 15 seconds, b, c, d 30 seconds, e, f, g 20 seconds
Seal&Protect [™] (Dentsply DeTrey, Konstanz, Germany; lot 0204001212)	Di- and trimethacrylate resins, PENTA, functionalized amorphous sicila, photo-initiators, butylated hydroxytoluence, cetilamide hydrofluoride. triclosan. acetone	a 15 seconds, b, c, d 10 seconds, g 10 seconds, e, f, g 20 seconds
Gluma Desensitizer™ (Heraeus Kulzer, Dormagen, Germany; lot 010032)	HEMA, glutaraldehyde, and purified water	a 15 seconds, b, c, d 30 seconds, e, f, g 20 seconds

a, acid etching; b, rinse; c, air dry; d, antibacterial primer; e, Transbond adhesive primer; f, Transbond adhesive paste; g, light cure. HEMA, 2-hydroxyethyl methacrylate; PENTA, pentaacryloyldipentaerythrythol phosphoric acid.



Figure 1 Universal testing machine including the orthodontic composite on the enamel surface.



Figure 2 Orthodontic composite block over enamel.

statistically significant differences in SBS between the various groups tested (P < 0.05).

The Mann–Whitney U analysis indicated that the highest SBS values were in the control (mean 35.15 ± 7.85 MPa) and Seal&ProtectTM (mean 27.98 ± 8.73 MPa) (P > 0.05) groups. On the other hand, when the Gluma DesensitizerTM and Micro PrimeTM were applied to the etched enamel surface, significantly lower SBS values were observed when compared with the control and Seal&ProtectTM groups (mean 21.61 ± 7.96 and 4.89 ± 5.55 MPa, respectively) (P < 0.05).

The fracture patterns of the specimens are shown in Table 3. In general, a greater percentage of the fractures were adhesive at the tooth–composite junction, except for Seal&ProtectTM (P < 0.05).

Discussion

The results of this study indicate that the application of Seal&ProtectTM containing triclosan on an etched enamel surface did not adversely affect the SBS of the adhesive, while the application of Gluma DesensitizerTM and Micro PrimeTM remarkably decreased bond strength.

Hahn *et al.* (1992) indicated that microbes accumulate on restorative materials. Among these, *mutans* streptococci



Figure 3 Application of force on the composite block with a stubbyshaped apparatus.

are known to cause caries at the margins of composite restorations as well as directly attacking the enamel (Shklair *et al.*, 1972; Svanberg *et al.*, 1990). The effects on adhesive bond strength of using fluoride solutions, gels, and rinses have been documented (Hirce *et al.*, 1980; Aboush *et al.*, 1991; Garcia-Godoy *et al.*, 1991a, b; Garcia-Godoy, 1993). The antibacterial effectiveness shown by the dental materials in some studies was related to either their pH or chemical composition (Imazato *et al.*, 2001). Current desensitizers include antibacterial components such as fluoride, triclosan, benzalkonium chloride, ethylene dianinetetraacetic acid, and glutaraldehyde (Duran *et al.*, 2003).

The antibacterial activity of Micro Prime[™] is related to the benzalkonium chloride content (Duran *et al.*, 2003; Sengün *et al.*, 2003), while that of Gluma Desensitizer[™] may be related to the glutaraldehyde content. Some other products that also contain glutaraldehyde have demonstrated antibacterial effectiveness, but these were shown to be dependent upon the leaching of glutaraldehyde from the cured materials (Fraga *et al.*, 1996; Meiers and Miller, 1996). In the present study, Gluma Desensitizer[™] was not cured before composite placement. The antibacterial activity of Seal&Protect[™] is related to the triclosan content. Little or no information is available on the use of liquid forms of antimicrobial agents after etching the enamel and before placing the bracket.

Many factors contribute to incomplete resin penetration of the enamel surface and the reduction in bond strength, including the layer of antibacterial agent applied blocking the enamel tags. The reduction noted with Gluma DesensitizerTM and Micro PrimeTM might be the result of the antibacterial agent in the product.

Table 2 Mean \pm standard deviation (MPa) of shear bond strength values and statistical comparison of groups (n = 18).

Groups	Mean ± standard deviation*	
Control Seal & ProtectTM	35.15 ± 7.85^{a}	
Gluma Desensitizer [™] Micro Prime [™]	$21.61 \pm 7.96^{\text{b}}$ $14.89 \pm 5.55^{\text{c}}$	

*According to the Mann–Whitney *U*-test adjusted by Bonferroni, means having the same letter are not statistically different from each other (P > 0.05).

 Table 3
 Modes of failure after shear bond testing.

Groups	Adhesive	Mixed
Control	17 (94.4%)	1 (5.6%)
Seal&Protect TM	13 (72.2%)	5 (27.8%)
Gluma Desensitizer TM	18 (100%)	0
Micro Prime™	17 (94.4%)	1 (5.6%)

Pearson chi-square = 9.336; P = 0.025.

Karaman and Uysal (2004) evaluated whether different types of antimicrobial agent with hydrophilic primer applied to etched enamel surfaces affect SBS and the bracket/ adhesive failure modes of metallic orthodontic brackets. In that study, teeth in the first group were used as a control and bonded with standard procedures. For the other three groups, mixtures containing a hydrophilic primer (Transbond MIP, 3M Unitek) and one of three antimicrobial agents were prepared: CervitecTM varnish (chlorhexidine and thymol in 1:2 ratio), chlorhexidine mouthwash (0.012 per cent chlorhexidine gluconate) and EC40[™] varnish (40 per cent chlorhexidine, sandarac, and ethanol in 1:1 ratio). These mixtures were applied to the etched enamel surfaces and light cured for 20 seconds. The brackets were then bonded and light cured for 40 seconds. The SBS values in these four groups compared favourably with those from other investigations and the minimal bond strength values were clinically acceptable. However, the results demonstrated that the control and CervitecTM varnish groups had higher SBS values than the other applications.

In the present investigation, composite specimens were used instead of brackets to test bond strength, as the bracket base design may contribute to the misalignment of load application during testing, making the bonding system prone to failure at the resin and enamel interface. It has also been found that variability exists among manufacturers with respect to the design or dimensions of the brackets in nominally identical prescriptions (Büyükyılmaz *et al.*, 1995). This variability poses a significant problem in studies evaluating bracket bond strength (Katona, 1997). Because the thickness of the adhesive layer is small, the tips of the SBS test blades cannot accurately be placed once the force is applied. The tips of the test blades may deviate towards either the joint between the adhesive and the bracket base or between the adhesive and the enamel, which may significantly affect the results. Blunting of the blades during use, particularly pointed ones, would have an increasing effect on the force level applied on later specimens (Arici and Minors, 2000). For these reasons, only orthodontic composite blocks without a bonded bracket were used for SBS testing.

Most orthodontic bonding studies have shown a mix or cohesive type failure (Årtun and Bergland, 1984; Oliver, 1988). In those studies, after SBS testing a part of the composite resin remained on either the enamel surface or the bracket base, causing cohesive failure rather than adhesive failure between the enamel and composite resin. Because brackets were not used in the present study, more adhesive failures occurred and the actual SBS between the enamel and composite could be measured. The higher percentage of adhesive failures also confirmed the accuracy of the SBS method.

Reynolds (1975) determined the minimum bond strength values in direct orthodontic bonding systems that are clinically acceptable to be 5.9–7.8 MPa. The bond strength values in the four groups in the present study compared favourably with those recommendations. However, clinical conditions may differ significantly from an *in vitro* setting. It needs to be emphasized that this was an *in vitro* study and the test conditions have not been subjected to the rigours of the oral environment (Bishara *et al.*, 1998). Heat and humidity conditions in the oral cavity are highly variable. Because of the probable differences between *in vivo* and *in vitro* conditions, as well as the method of testing, a direct comparison cannot be made with the findings of other studies.

The application of antibacterial agents would seem to be a suitable chairside technique without any significant difference in bond strength.

Conclusions

From the results of this study, it can be concluded that the application of Seal&ProtectTM containing triclosan to the etched enamel surface did not cause a significant decrease in bond strength. However, Gluma DesensitizerTM and Micro PrimeTM resulted in a decrease in bond strength to the etched enamel surface. These results need to be confirmed clinically.

Further clinical investigations are also required to test whether these antibacterial primers can prevent white spot lesions or dental caries.

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