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Evaluation of shear bond strength with different enamel pre-treatments

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SUMMARY The purpose of this study was to investigate the shear bond strengths of two adhesives, Panavia-21 and a composite resin (Transbond XT), with different enamel pre-treatments, acid etching (37 per cent phosphoric acid) and grit blasting (50 μm aluminium oxide particles). The mode of bond failure was also assessed using the modified adhesive remnant index (ARI). Ninety freshly extracted non-carious human premolar teeth were randomly divided into the following groups: (1) Transbond XT, acid-etched enamel surface; (2) Panavia-21, acid-etched enamel surface; (3) Transbond XT, grit-blasted enamel surface; (4) Panavia-21, grit-blasted enamel surface; (5) Transbond XT, acid-etched enamel surface with grit-blasted brackets; (6) Panavia-21, acid-etched enamel surface with grit-blasted brackets. All groups had stainless steel brackets bonded to the buccal surface of each tooth. An Instron universal testing machine was used to determine the shear bond strengths at a crosshead speed of 0.5 mm/second. Statistical analysis was undertaken using analysis of variance and the Tukey test.

The mean bond strength values were as follows: group 1, 135.7 ± 23.0 N; group 2, 181.5 ± 18.4 N; group 3, 38.4 ± 27.5 N; group 4, 59.1 ± 24.1 N; group 5, 106.7 ± 21.5 N; group 6, 165.3 ± 21.4 N. Panavia-21 with the acid-etched enamel surface had a significantly higher shear bond strength than the other groups ($P < 0.001$). This was followed by the composite group with the acid-etched enamel surface. This group differed significantly from the composite and Panavia-21 groups with the grit-blasted tooth surface ($P < 0.001$) and from the composite and Panavia-21 groups with the acid-etched enamel surface and grit-blasted brackets ($P < 0.01$).

The current findings indicate that Panavia-21 is an excellent adhesive and produces a bond strength that is clinically useful. Enamel surface preparation using grit blasting alone results in a significantly lower bond strength and should not be advocated for clinical use.

Introduction

Panavia (a modified phosphate ester Bis-GMA composite) has recently been investigated for use in orthodontics (Rux *et al.*, 1991; Ireland and Sherriff, 1994; McSherry, 1996). It contains a special monomer which has a phosphate group in its molecular structure (Omura *et al.*, 1984) and develops an acceptable bond strength to both tooth structure and grit-blasted non-precious alloy (Saunders, 1986; Creugers *et al.*, 1988).

The acid etch bonding technique requires the creation of microporosities in enamel surfaces that result in a micromechanical bond. It is commonly assumed that to obtain a good bond strength and minimal enamel loss, the enamel must be etched for 15 seconds with 35–40 per cent phosphoric acid (Sheen *et al.*, 1993; Wang *et al.*, 1994).

The air abrasion technique (grit blasting) is another method of enamel pre-treatment. It makes use of a high speed spectrum of aluminium oxide particles (50 μm) propelled by air pressure. Air abrasion technology has been advocated for the preparation of enamel to obtain micromechanical bonding (Goldstein and Parkins, 1994). It has been suggested that, because the air abrasion technique roughens the enamel surface, it could possibly provide direct bonding to orthodontic brackets without acid etching (Keen *et al.*, 1994; Reisner *et al.*, 1995).

The purpose of this study was to compare the shear bond strengths of Panavia (Panavia-21, Kurary-Dental, Morita, USA) and a composite resin control (Transbond XT, 3M/Unitek, Monrovia, California, USA) with traditional acid etching and the air abrasion technique.

Materials and methods

Teeth

Upper and lower premolar teeth ($n = 90$) were collected from patients having extractions for orthodontic reasons and stored in 10 per cent thymol solution at room temperature. The buccal crown surface of each tooth was examined under ×10 magnification to ensure that it was free of caries, restorations or cracks, which might affect their resistance to experimental loading.

Bonding

The 90 teeth were randomly divided into six groups each containing 15 teeth. Each tooth was mounted in

cold-curing, fast-setting acrylic (Leocryl; Leone, Sesto Fiorentino, Italy). The teeth were aligned so that their labial surface was exposed and paralleled the force during shear bond testing. The buccal surface of each tooth was polished with fluoride-free pumice slurry for 15 seconds, rinsed and dried. Standard edgewise metal premolar brackets (mesh backed, 0.022 inch Roth prescription without hook; Leone) were bonded to the teeth with a different adhesive or protocol used for each group. The composite resin (Transbond XT) was light cured (XL300; 3M/Unitek).

Group 1: Transbond XT, acid-etched enamel surface (control group)

The buccal enamel surface was etched with 37 per cent phosphoric acid for 15 seconds, rinsed for 15 seconds, and dried with oil-free air until the enamel had a white appearance. Transbond XT primer was applied to the etched surface in a thin film. Transbond XT adhesive paste was applied to the bracket base and the bracket was positioned and pressed firmly onto the tooth. The excess adhesive was removed from around the bracket base and the adhesive was light cured for 40 seconds.

Group 2: Panavia-21, acid-etched enamel surface

The buccal enamel surface was prepared as for group 1. Panavia-21 primer was applied to the etched surface in a thin film. Panavia-21 paste was applied to the bracket base and the bracket was positioned and pressed firmly onto the tooth. The excess adhesive was removed from around the bracket base. As Panavia-21 has anaerobic curing properties and should be isolated from oxygen in the air to polymerize fully, the brackets were coated with an oxyguard for 2 minutes to prevent oxidation and allow setting of the adhesive.

Group 3: Transbond XT, grit-blasted tooth surface

Grit blasting was undertaken with 50 μm aluminium oxide at 80 psi for 5 seconds at a nozzle distance of 10 mm and an angle of 45 degrees. After grit blasting, the buccal enamel surface was cleaned with oil-free compressed air. Transbond XT primer was applied to the abraded surface in a thin film. Transbond XT adhesive paste was applied to the bracket base and the bracket was positioned and pressed firmly onto the tooth. The excess adhesive was removed from around the bracket base and the adhesive was light cured for 40 seconds.

Group 4: Panavia-21, grit-blasted enamel surface

The surface was prepared as for group 3. Panavia-21 primer was applied to the etched surface in a thin film. Panavia-21 paste was applied to the bracket base and the bracket was positioned and pressed firmly onto the tooth. The excess adhesive was removed from around the bracket base. The bracket was coated with oxyguard for 2 minutes to prevent oxidation and allow setting of the adhesive.

Group 5: Transbond XT, acid-etched enamel surface, grit-blasted bracket base

The bracket base was grit blasted with 50 μm aluminium oxide at 80 psi for 15 seconds at a nozzle distance of 1 mm. The buccal enamel surface was etched with 37 per cent phosphoric acid for 15 seconds, rinsed for 15 seconds, and dried with oil-free air until the enamel had a white appearance. Transbond XT primer was applied to the etched surface in a thin film. Transbond XT adhesive paste was applied to the bracket base and the bracket was positioned and pressed firmly onto the tooth. The excess adhesive was removed from around the bracket base and the adhesive was light cured for 40 seconds.

Group 6: Panavia-21, acid-etched enamel surface, grit-blasted bracket base

The bracket base was prepared as for group 5. Panavia-21 primer was applied to the etched surface in a thin film. Panavia-21 paste was applied to the bracket base and the bracket was positioned and pressed firmly onto the tooth. The excess adhesive was removed from around the bracket base. The bracket was coated with oxyguard for 2 minutes to prevent oxidation and allow setting of the adhesive.

After bonding all samples were stored in distilled water at room temperature for 24 hours and subsequently tested in a shear mode on a universal testing machine (Instron 1195, Instron Limited, High Wycombe, UK) (Figure 1). For shear testing, the specimens were secured in the lower jaw of the machine so that the bracket base paralleled the direction of the shear force. The force required to debond was measured in Newtons (N) at a crosshead speed of 0.5 mm/second.

After bond failure the bracket bases and enamel surfaces were examined by the same operator under a light stereomicroscope at ×10 magnification. The amount of adhesive left on the enamel surface was scored for each tooth using the modified adhesive remnant index (ARI) (Oliver, 1988). The ARI scale ranges from 1 to 5: 1, all adhesive remaining on the enamel surface, with the impression of the bracket base; 2, more than 90 per cent of the adhesive remaining on the enamel surface; 3, less than 90 per cent but more than 10 per cent of the adhesive remaining on the enamel surface; 4, less than 10 per cent of the adhesive remaining on the enamel surface; 5, no adhesive remaining on the enamel surface.

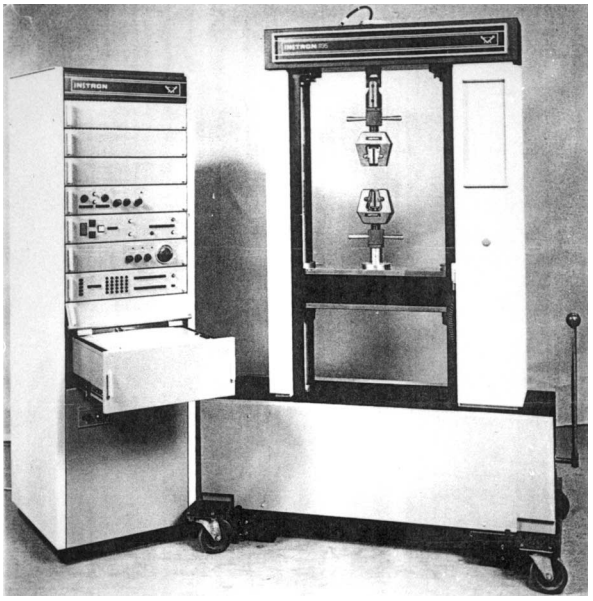

Figure 1 The Instron model 1195 testing instrument.

Method error

Twenty randomly selected teeth were re-examined on two occasions separated by a period of 1 week, and the kappa test was used to test intra-examiner reliability. Kappa values ranged between 96 and 100 per cent for the ARI scores.

Statistical analysis

Descriptive statistics, including the mean, standard deviation and 95 per cent confidence intervals, were calculated for each of the six groups. The analysis of variance was used to determine whether significant differences existed between the groups. If a significant difference was present, Tukey multiple comparison tests were used to identify which of the groups were different. The chi-squared test was used to determine significant differences in the ARI scores between the groups. Significance for all statistical tests was predetermined at $P < 0.05$.

Results

The mean, standard deviations and 95 per cent confidence intervals for the shear bond strengths in the six groups are shown in Table 1 and Figure 2.

Acid-etched enamel surface groups

The shear bond strength was significantly greater in the Panavia-21 group ($P < 0.001$). It averaged 181.5 ± 18.4 N. In the Transbond XT group, the shear bond strength was 135.7 ± 23.0 N.

Scanning electron micrographs (SEMs) of the bracket bases of the Transbond XT and Panavia-21 groups are shown in Figure 3a and b, respectively.

Grit-blasted enamel surface groups

The shear bond strengths in the groups with grit-blasted tooth surfaces were significantly lower than the other groups ($P < 0.001$). They averaged 59.1 ± 24.1 and 38.4 ± 27.5 N for the Panavia-21 and Transbond XT groups, respectively. The difference between the two groups was significant ($P < 0.05$).

SEMs of the bracket bases of the Transbond XT and Panavia-21 groups are shown in Figure 4a and b, respectively.

Acid-etched enamel surface with grit-blasted brackets groups

The shear bond strengths in this group were significantly lower than in the groups with etched enamel and without grit-blasted brackets. The shear bond strengths for the Panavia-21 and Transbond XT groups were 165.3 ± 21.4 and 106.7 ± 21.5 N, respectively. The difference between the two groups was significant ($P < 0.001$).

SEMs of the bracket bases of the Transbond XT and Panavia-21 groups are shown in Figure 5a and b, respectively.

ARI

The ARI scores for the six groups tested are shown in Table 2. The results indicated the presence of a significant difference between the groups ($P < 0.001$) (Table 3).

Table 1 Means, standard deviations (SD) and 95 per cent confidence intervals (95% CI) for the shear bond strengths of the tested groups.

Group	Material	Enamel surface pre-treatment	Brackets	Mean ± SD (N)	95% CI
1	Transbond XT	Acid etched	–	135.7 ± 23.0	122.9 – 148.5
2	Panavia-21	Acid etched	–	181.5 ± 18.4	171.3 – 191.6
3	Transbond XT	Grit blasted	–	38.4 ± 27.5	23.2 – 53.6
4	Panavia-21	Grit blasted	–	59.1 ± 24.1	45.7 – 72.4
5	Transbond XT	Acid etched	Grit blasted	105.4 ± 23.3	98.0 – 113.9
6	Panavia-21	Acid etched	Grit blasted	165.3 ± 21.4	153.5 – 177.2

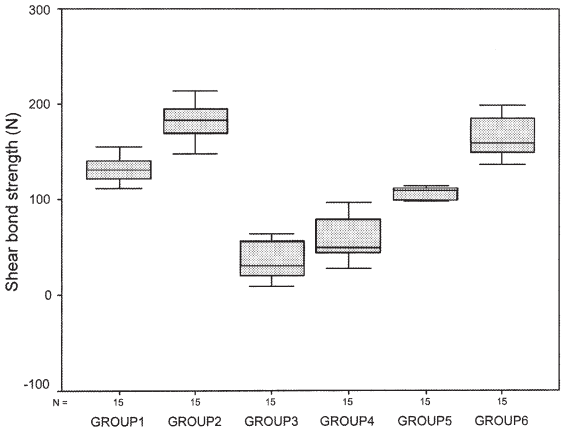

Figure 2 The mean shear bond strength, standard deviation and 95 per cent confidence interval for each of the six groups.

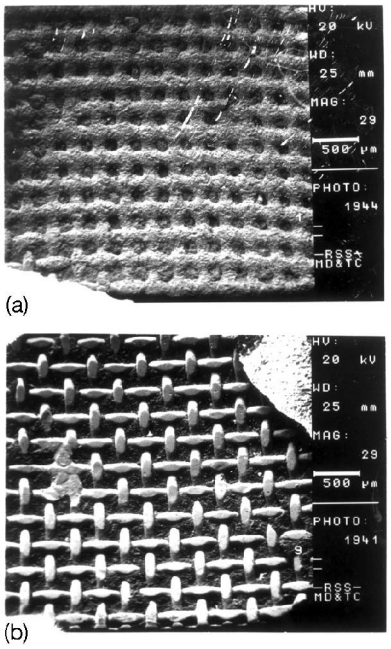

Figure 3 Debonded bracket surfaces of teeth prepared with acid etching using (a) Transbond XT and (b) Panavia-21.

Examination under the light stereomicroscope at ×10 magnification revealed that the grit-blasted tooth groups had greater ARI scores of 4 and 5. This indicated that bond failure in these groups occurred more frequently at the enamel–adhesive interface. For the grit-blasted brackets with acid-etched enamel surfaces, an ARI score of 3 was most frequent.

For the acid-etched enamel surface without grit blasting the brackets, ARI scores of 1 and 2 were most frequent, indicating that bond failure in these groups occurred at the bracket–adhesive interface.

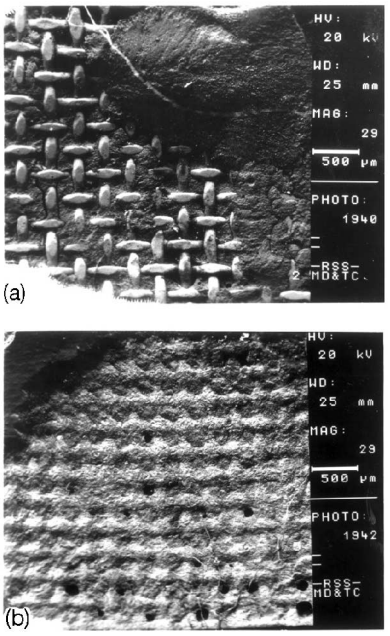

Figure 4 Debonded bracket surfaces of teeth prepared with grit blasting only using (a) Transbond XT and (b) Panavia-21.

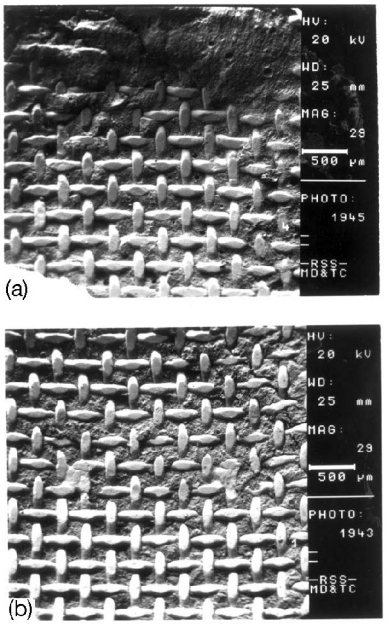

Figure 5 Debonded bracket surfaces of teeth prepared with acid etching and grit blasting the bracket surface using (a) Transbond XT and (b) Panavia-21.

Discussion

Two methods have been described to treat enamel surfaces before bonding the adhesive, namely, acid etching (Newman, 1965) and air abrasion (Katora *et al.*, 1981). Acid etching produces sufficient bond strength

Table 2 Adhesive remnant index (ARI) scores for the groups tested.

Group	ARI scores				
	1	2	3	4	5
Transbond XT, acid-etched enamel surface	8	2	5	–	–
Panavia-21, acid-etched enamel surface	5	6	4	–	–
Transbond XT, grit-blasted enamel surface	–	–	1	3	11
Panavia-21, grit-blasted enamel surface	–	–	5	9	1
Transbond XT, acid-etched enamel surface, grit-blasted brackets	–	1	8	5	1
Panavia-21, acid-etched enamel surface, grit-blasted brackets	–	4	10	1	–

$\chi^2 = 117.5$, $P < 0.001$.

Table 3 Comparison of adhesive remnant index (ARI) scores using the Tukey test.

Group	ARI scores					
	1	2	3	4	5	6
Transbond XT, acid-etched enamel surface	_	***	***	***	**	**
Panavia-21, acid-etched enamel surface	***	_	***	***	***	NS
Transbond XT, grit-blasted enamel surface	***	***	_	*	***	***
Panavia-21, grit-blasted enamel surface	***	***	*	_	***	***
Transbond XT, acid-etched enamel surface, grit-blasted brackets	**	***	***	***	_	***
Panavia-21, acid-etched enamel surface, grit-blasted brackets	**	NS	***	***	***	_

NS, not significant; *$P < 0.05$; **$P < 0.01$; ***$P < 0.001$.

but causes damage to the enamel surface (Diedrich, 1981; Barkmeier *et al.*, 1985). The loss of enamel substance is not irreversible, and remineralization can occur (van Waveren Hogervorst *et al.*, 2000). It varies according to the exposure times and the acid concentrations used (Barkmeier *et al.*, 1985; Wang *et al.*, 1994). Wang *et al.* (1994) recommended the use of phosphoric acid (35–40 per cent) for 15–60 seconds to etch the enamel surface before bonding orthodontic brackets.

The air abrasion technique results in irreversible loss of enamel surface (Olsen *et al.*, 1997). However, if grit blasting is carried out at low pressure and for a short period of time, enamel loss will be reduced (Reisner *et al.*, 1997; van Waveren Hogervorst *et al.*, 2000). The above variables can be controlled by the operator.

Tavas and Watts (1984) suggested that a minimum bond strength of 58 N was adequate for clinical orthodontic bonding. Using the air abrasion technique for both Panavia-21 and Transbond XT, a shear bond strength which is lower than that recommended for clinical use was produced. This is in agreement with the findings of Olsen *et al.* (1997), Canay *et al.* (2000) and van Waveren Hogervorst *et al.* (2000).

McSherry (1996) evaluated the shear bond strength of Panavia and suggested that Panavia-Ex with grit-blasted brackets has the highest shear bond strength among the adhesives tested (101.5 ± 14.5 N). In the present study, Panavia with the acid-etched enamel surface had the highest shear bond strength. However, when the brackets were grit blasted the bond strength was reduced, but not to a statistically significant level. The reduction in bond strength may be caused by air entrapment in the grit-blasted mesh brackets, which affects setting of the anaerobic cement.

Transbond XT with the acid-etched enamel surface showed an adequate shear bond strength that was reduced significantly when the brackets were grit blasted. Grit blasting the mesh brackets may have reduced the mechanical lock between the adhesive and the bracket base and resulted in a reduced bond strength. However, this needs further investigation using brackets with a non-meshed surface.

Evaluation of the ARI scores indicated that there was a significantly higher frequency of bond failure at the bracket–adhesive interface in the acid-etched groups, whereas the grit-blasted enamel surface groups showed a significantly higher frequency of bond failure at the enamel–adhesive interface.

Assessment of enamel treatment before bonding suggests that compared with the grit-blasting technique the best bond strengths are obtained by etching the enamel surface.

Conclusions

1. Panavia-21 with the acid-etched enamel surface produced the highest shear bond strength. This was reduced when the brackets were grit blasted.
2. The shear bond strength of Transbond XT with the acid-etched enamel was reduced when the brackets were grit blasted.
3. Bond failure frequently occurred at the bracket–adhesive interface in the acid-etched enamel surface group and at the enamel–adhesive interface in the grit-blasted enamel group.

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