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The *in vitro* effect of repeated bonding on the shear bond strength with different enamel conditioning procedures

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SUMMARY The aims of this study were to evaluate the effect on shear bond strength (SBS), adhesive remnant, and enamel surface of repeated bonding of new brackets on the same tooth using different methods of enamel conditioning.

One hundred and thirty-five bovine incisors were used. Brackets were bonded to enamel using one of the following conditioning procedures: (1) 37 per cent phosphoric acid, (2) 37 per cent phosphoric acid (prior to first bond but not for further bonds), (3) Transbond Plus Self Etching Primer® (TSEP), and (4) non-rinse-conditioner (NRC). Brackets were sequentially bonded and debonded three times following the same conditioning procedure with the exception of group 2 where 37 per cent phosphoric acid was not reapplied prior to the second and third bonding sequences. SBS and adhesive remnant were evaluated for each debond. Scanning electron microscopy observations were made for each conditioning sequence. Statistical analysis was undertaken using ANOVA, Mann–Whitney, and Kruskal–Wallis tests.

Bond strength and adhesive remnant values were similar across the four groups for the first and second bonding sequences. At the third sequence, SBS was significantly less ($P < 0.008$) for group 2 (5.71 ± 1.56 MPa) than for group 1 (9.42 ± 2.75 MPa) and the adhesive remnant was significantly lower ($P < 0.008$) for group 2 (6.93% ± 3.34) than for the other groups (group 1: 16.95 ± 4.99 per cent, group 3: 14.40 ± 5.11 per cent, and group 4: 14.60 ± 5.33 per cent). When comparing the SBS and adhesive remnant of the three bonding/debonding sequences within each group, both the SBS and adhesive remnant for group 2 (SBS: 5.71 ± 1.56 MPa and adhesive remnant: 6.93 ± 3.34%) at the third sequence were significantly less ($P < 0.017$) than at the first (SBS: 10.44 ± 3.55 MPa and adhesive remnant: 13.81 ± 5.59%) and second (SBS: 9.23 ± 2.69 MPa and adhesive remnant: 15.32 ± 6.85%) sequences. Enamel changes were similar across all groups.

TSEP and NRC produced bonds that were similar to acid etching. When acid etching is used, it is possible to avoid etching for a second bond but not for following bonds.

Introduction

One of the most frequent problems facing the orthodontic clinician is bracket bond failure (Murfitt *et al.*, 2006; Banks and Thiruvenkatachari, 2007; Elekdag-Turk *et al.*, 2008), usually due to the patient applying excessive masticatory force, an inappropriate bonding technique or contamination of the tooth area during bonding (Donker *et al.*, 2001; Thiyagarajah *et al.*, 2006). Sometimes, the practitioner debonds a bracket in order to reposition it more appropriately to achieve a better outcome.

Whatever the cause of bond failure, repeated bonding on the same tooth involves both the removal of the bond material remaining on the tooth (Hong and Lew, 1995) and the repetition of acid etching. Both procedures are accompanied by some loss of the fluoride-rich surface enamel (Campbell, 1995; Tüfekçi *et al.*, 2004; Kim *et al.*, 2007).

Bonding brackets directly onto tooth enamel became possible with the introduction of the acid-etching technique developed by Buonocuore (1955). Since then, refinements of this technique have aimed to reduce both chair time and possible enamel loss. Weaker concentrations of phosphoric acid have been used and application time has been reduced (Kinch *et al.*, 1988), and some authors have proposed the use of other acids for etching: maleic (Urabe *et al.*, 1999; Bishara *et al.*, 2001) or nitric (Gardner and Hobson, 2001). At present, the most widely accepted etching technique is the application of phosphoric acid at 37 per cent for 30 seconds, although some authors argue that a 15 second application is sufficient (Wang and Yeh, 1994).

Recently, new alternatives to established acid-etch techniques have been introduced. Among these are non-rinse conditioners (NRCs), which condition the enamel without the need for washing, and self-etching primers (SEPs), which etch and prime in a single procedure. Various studies corroborate the efficacy of NRCs (Vicente *et al.*, 2005a) and SEPs (Vicente *et al.*, 2005b; Bishara and Ostby, 2006, 2007; Attar *et al.*, 2007; Davari *et al.*, 2007; Kitayama *et al.*, 2007) for bracket bonding, but research involving the use of these materials for successive rebonding is scarce. In fact, the use of NRCs for successive rebonds has not been evaluated, and only few studies exist on the use of SEPs (Hirani and Sherriff, 2006; Montasser *et al.*, 2008a,b) for repeat bonding. Furthermore, there are no studies to date that evaluate the effect of avoiding acid etching for successive rebonds when enamel has been conditioned with phosphoric acid prior to the initial bond.

The aims of this study were to evaluate the effect (1) on shear bond strength (SBS), (2) on the percentage of the area occupied by adhesive remaining on the teeth after debonding, and (3) on the structural changes to the enamel surface after carrying out successive rebonds with new brackets on the same tooth using four enamel conditioning methods. The null hypothesis tested is that after carrying out successive rebonds with new brackets on the same tooth using four enamel conditioning methods, there are no significant differences in SBS, on the percentage of area occupied by adhesive remaining on teeth after debonding, and on the structural changes to the enamel surface.

Material and methods

Teeth

One hundred and thirty-five bovine upper central incisors visually intact and with no cracks on the enamel surface were used. The teeth were washed in water to remove any traces of blood and then placed in a 0.1 per cent thymol solution. They were then stored for a period of less than 1 month in distilled water, which was changed daily to avoid deterioration.

For SBS testing, 80 teeth were used; they were set in a 4 cm long copper cylinder with an internal diameter of 3 cm, with their roots in Type IV plaster. Fifty-five teeth were used for scanning electron microscopy (SEM) observations.

Brackets

Three hundred upper central incisor brackets (Victory Series®, 3M Unitek Dental Products, Monrovia, California, USA) were used, of which 240 were used for SBS testing and 60 for SEM observations. The base area of each bracket was calculated (mean = 10.25 mm^2) using image analysis equipment and MIP 4 software (Microm Image Processing Software, Digital Systems, Barcelona, Spain).

Bonding procedure

Eighty teeth were divided into the equal four groups and brackets were bonded on the buccal surfaces according to the manufacturer's instructions. For all groups, the buccal surfaces were polished with a rubber cup and polishing paste (Détartrine®, Septodont, Saint-Maur, France).

Group 1 (n = 20): the buccal surfaces were etched with 37 per cent *o*-phosphoric acid gel (Total Etch, Ivoclar, Vivadent, Schaan, Liechtenstein) for 30 seconds. The enamel was then washed with water and dried with compressed oil-free air. A layer of Transbond XT® primer (3M Unitek Dental Products) was applied to the tooth. Transbond XT® paste was applied to the base of the bracket and pressed firmly onto the tooth. Excess adhesive was removed from around the base of the bracket and the adhesive was light-cured, positioning the light guide of an Ortholux XT® lamp (3M Unitek Dental Products) on each interproximal side for 10 seconds.

Group 2 (n = 20): the brackets were bonded as in group 1.

Group 3 (n = 20): the enamel was treated with Transbond Plus Self-Etching Primer® (TSEP), 3M Unitek Dental Products, which was gently rubbed onto the enamel for 5 seconds with the disposable applicator supplied by the manufacturer. A moisture-free air source was used to deliver a gentle burst of air to the primer. The bracket was bonded with Transbond XT paste as in group 1.

Group 4 (n = 20): the enamel was treated with a NRC (Dentsplay de Trey, Konstanz, Germany). NRC was gently brushed onto the enamel leaving it undisturbed for 20 seconds. A moisture-free air source was then used to deliver a gentle burst of air to the enamel. The bracket was then bonded with Transbond XT (primer and paste) as in group 1.

Storage of test specimens

The specimens were immersed in distilled water at a temperature of 37°C for 24 hours.

Bond strength test

SBS was measured with a universal testing machine (Autograph AGS-1KND, Shimadzu, Kyoto, Japan) with a 1 KN load cell connected to a metal rod with one end angled at 30 degrees. The crosshead speed was 1 mm/minute.

The teeth were set at the base of the machine so that the sharp end of the rod incised in the area between the base and the wings of the bracket, exerting a force parallel to the tooth surface in an inciso-apical direction.

The force required to debond each bracket was registered in Newtons (N) and converted into megapascals (MPa) as a ratio of Newtons to surface area of the bracket (MPa = N/mm^2).

Repeated bonding

After debonding, the remaining bond material was removed from the enamel surface with a finishing carbide bur (Komet FG H22 GK016, Besigheim, Germany). Bonding/debonding procedures were repeated three times for each tooth. A new bracket was used for each successive bond procedure. Brackets were bonded following the same procedures as described for each group with the exception of group 2 in which for the second and third bonds, phosphoric acid was not used and Transbond XT® primer was applied directly to the enamel.

Evaluation of residual adhesive

The percentage of the surface of the bracket base covered by adhesive after debonding was determined using image analysis equipment (Sony dxc 151-ap video camera, connected to an Olympus SZ11 microscope) and MIP 4 software.

The percentage of the area occupied by adhesive remaining on the tooth after debonding was obtained by subtracting the area of adhesive covering the bracket base from 100 per cent.

Statistical analysis

SBS values and the data for the percentage of the area occupied by adhesive remaining on the teeth after debonding were compared for the four bonding procedures at each bonding/debonding sequence. Comparisons were also performed to determine whether significant differences existed in SBS and the percentage of area of adhesive remaining on teeth between the three bonding/debonding sequences within each bonding procedure.

Kolmogorov–Smirnov's normality and Levene's homogeneity of variance tests were applied to the data for bond strength and percentage of area of adhesive remaining on the teeth after debonding. When the data fulfilled the criteria for normality and homogeneity of variance, the existence of significant differences was determined by means of variance analysis (ANOVA) for one factor and Scheffé test for multiple comparisons ($P < 0.05$). When the data were not normally distributed or failed to fulfil the criteria for variance homogeneity, it was analysed using the Kruskal–Wallis test ($P < 0.05$), finding those groups that were significantly different with the Mann–Whitney test for two independent samples. In order to avoid an accumulation of errors due to multiple comparisons, the significance level was modified dividing this ($P < 0.05$) by the number of comparisons made (Bonferroni correction). $P < 0.017$ was considered significant when three comparisons were made and $P < 0.008$ for six comparisons.

SEM observation

The conditioned and reconditioned enamel surfaces of 55 teeth were observed after the brackets had been debonded following the procedures described above. The same procedures were used for bond strength testing, except that brackets were debonded using debonding pliers (Leone P 1920-00 CE, Florence, Italy).

Teeth treated with TSEP were rinsed with acetone for 10 seconds to remove the SEP (Kanemura *et al.*, 1999). Afterwards, the teeth were cleaned in distilled water with ultrasonic agitation for 30 minutes and gently air-dried. They were then fixed to SEM stubs, sputter coated with gold, and examined under a Jeol-6100 (Tokyo, Japan) scanning eletron microscope operating at 15kV. Representative images for each different surface treatment were captured and stored digitally.

Results

No significant differences were observed between the four procedures at the first ($P = 0.06$) or second ($P = 0.05$) bonding sequences. However, at the third sequence, the SBS in group 2 was significantly less than in group 1 ($P = 0.000$) (Table 1). For the percentage of tooth occupied by bond material remnant after debonding, the data showed no significant differences between the four bond procedures at the first ($P = 0.15$) or second ($P = 0.28$) sequence, while at the third sequence, the percentage of tooth area occupied was significantly less in group 2 than in the other groups (group 1 $P = 0.000$, group 3 $P = 0.000$, and group 4 $P = 0.000$) (Table 2).

When comparing the SBS of the three bonding/debonding sequences within each bonding procedure, only group 2 ($P = 0.00$) showed significant differences, bond strength at the third sequence being significantly less than at the first ($P = 0.000$) and second ($P = 0.000$) sequence. No significant difference was found between the first and second sequences ($P = 0.273$) for this group. For the other groups evaluated, there were no significant differences between the three bonding/debonding sequences (group 1 $P = 0.46$, group 3 $P = 0.94$, and group 4 $P = 0.77$) (Table 1). Data for the percentage of area occupied by adhesive did not show significant differences between the three bonding/debonding sequences for groups 1 ($P = 0.25$), 3 ($P = 0.06$), and 4 ($P = 0.84$). However, in group 2 at the third sequence, the area occupied by adhesive was significantly less than at the first ($P = 0.000$) and second ($P = 0.000$) sequences. No significant

Table 1 Mean shear bond strength (MPa) and standard deviation (SD) for group 1: 37 per cent phosphoric acid; group 2: 37 per cent phosphoric acid only etched for the first bond, not for further bonds; group 3: Transbond self-etching primer; and group 4: non-rinse conditioner, after repeated bonding and debonding ($n = 20$).

Debonding sequence	Group			
	1	2	3	4
	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD
1	10.50 ± 3.73	10.44 ± 3.55A	8.19 ± 3.19	8.07 ± 2.73
2	10.13 ± 3.61	9.23 ± 2.69A	7.60 ± 1.12	7.97 ± 2.20
3	9.42 ± 2.75a	5.71 ± 1.56Bb	7.85 ± 3.12	7.14 ± 2.62

Different upper case letters within the same column indicate significant differences ($P < 0.017$). For each row, different lower case letters indicate significant differences ($P < 0.008$). Within the same column or the same row, the values unmarked by upper or lower case letters did not show significant differences with any other ($P > 0.05$).

differences were found between the first and second sequences ($P = 0.429$) (Table 2).

When enamel surfaces were examined under SEM after the first conditioning, the presence of porosities in the enamel could be seen in all groups, although the etching pattern for the NRC resulted in improved enamel conservation. After the second and third sequences, composite remnants were observed on the enamel surfaces in all groups (Figure 1).

Discussion

Bovine incisors were used in this study because of their microstructural similarity to human tooth enamel (Oesterle *et al.*, 1998) and also because of the easy availability of samples with intact vestibular surface enamel.

Various authors (Buchman, 1980; Egan *et al.*, 1996; Grabouski *et al.*, 1998; Chung *et al.*, 2000; Tavares *et al.*, 2006) have evaluated SBS obtained after rebonding reused brackets on teeth that had not undergone previous bonding. However, there are only a limited number of studies that have evaluated the rebonding of new brackets on the same tooth surface (Bishara *et al.*, 2000; Montasser *et al.*, 2008a,b).

No significant differences in bond strength between the different enamel conditioning procedures for the first bond/debond sequence were found in the present study. In agreement with Vicente *et al.* (2006), the NRC resulted in bond strength values similar to those obtained with phosphoric acid. For the SEPs, the results agree with other studies in which the bond strength values obtained with these materials were similar to those with the traditional technique (Hirani and Sherriff, 2006; Holzmeier *et al.*, 2008; Iijima *et al.*, 2008; Montasser *et al.*, 2008a,b). However, some authors have reported bond strength values, which were significantly less with SEPs than with phosphoric acid (Bishara *et al.*, 2001; Aljubouri *et al.*, 2003).

No significant differences were observed in bond strength between the four conditioning methods at the second bond/debond sequence. The fact that the bond strength in group 1 was similar to that obtained in group 2 in which brackets were rebonded without a second etching is interesting clinically, given that avoiding acid etching will have the advantage of reducing chair time as well as enamel loss. Surface enamel loss during acid etching is estimated at between 10 and 30 μm (Wickwire and Rentz, 1977). However, at the third debond, there was a significant difference in bond strength between the no prior acid-etch group (group 2) and the phosphoric acid-etch group (group 1), suggesting that when brackets are bonded for a third time on the same tooth, it would seem advisable to apply phosphoric acid again in order to achieve an acceptable bond strength. NRC and TSEP achieved similar bond strengths at the third bond/debond sequence to the acid-etch procedure.

Table 2 Mean and standard deviation (SD) of the percentage of tooth area occupied by adhesive for group 1: 37 per cent phosphoric acid; group 2: 37 per cent phosphoric acid only etched for the first bond, not for further bonds; group 3: Transbond self-etching primer; and group 4: non-rinse conditioner, after repeated bonding and debonding ($n = 20$).

Debonding sequence	Group			
	1	2	3	4
	Mean ± SD	Mean ± SD	Mean ± SD	Mean ± SD
1	14.34 ± 2.46	13.81 ± 5.59Aa	16.21 ± 7.80	13.71 ± 5.28
2	17.98 ± 8.45	15.32 ± 6.85Aa	17.00 ± 5.82	14.09 ± 4.01
3	16.95 ± 4.99a	6.93 ± 3.34Bb	14.40 ± 5.11a	14.60 ± 5.33a

Different upper case letters within the same column indicate significant differences ($P < 0.017$). For each row, different lower case letters indicate significant differences ($P < 0.008$). Within the same column or the same row, the values unmarked by upper or lower case letters did not show significant differences with any other ($P > 0.05$).

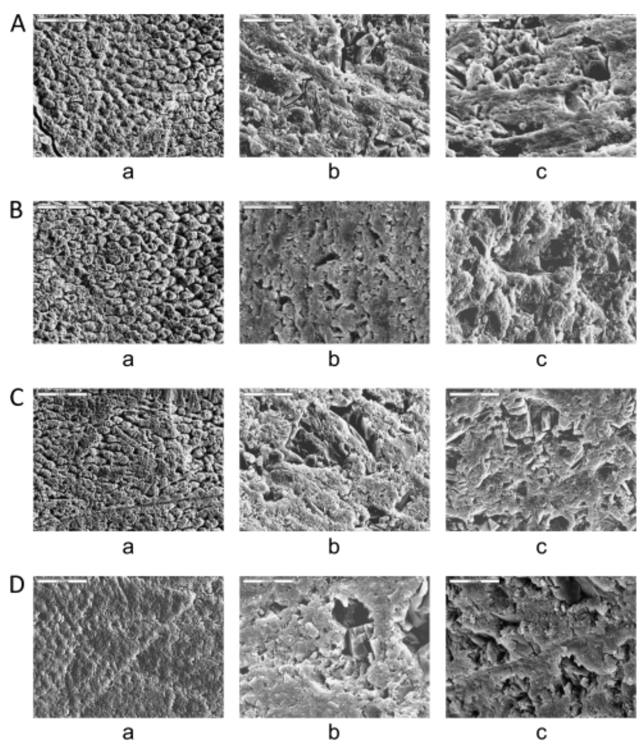

Figure 1 Scanning electron microphotographs of (A) group 1 (37 per cent phosphoric acid), (B) group 2 (37 per cent phosphoric acid only etched for the first bonding sequence, not for further bonds), (C) group 3 (Transbond self-etching primer), and (D) group 4 (non-rinse conditioner) for the first (a), second (b), and third (c) bond/debond sequences (×1500, bar = 20 μm).

When bond strength values for each conditioning procedure were compared at the three bond/debond sequences, a general reduction in SBS at each repetition was seen. However, no significant differences were observed for the NRC, the SEP, or the group in which acid etching was applied prior to each bond.

The present results do not agree with Bishara *et al.* (2000), who, after etching with phosphoric acid for all bond/debond sequences, concluded that rebonded teeth have significantly lower and inconsistent SBS. This may be due to the different methodologies used and the fact that in the earlier study bond strength testing took place 30 minutes after bonding and the machine crosshead speed was 5 mm/minute.

For the self-etching adhesive, the present results were similar to those of Montasser *et al.* (2008a,b) who found that with repeated bonding/debonding, bond strength did not differ significantly.

In group 2, which did not undergo acid etching at the second and third bond/debond sequences, similar bond strengths were achieved as in the first sequence (that had been etched with phosphoric acid) and the second sequence. However, bond strength at the third sequence was significantly less than that at the first and second sequences. This result highlights the convenience of acid etching when bonding for a third time.

When the area occupied by adhesive remaining on the teeth after debonding was measured, no significant differences were found between the different conditioning procedures at either the first or second bond/debond sequence. However, at the third sequence, there was significantly less adhesive left on teeth in group 2, which had not been acid etched prior to bonding compared with the other groups (1, 3, and 4). Within each conditioning procedure, the adhesive remaining was similar for all three sequences, with the exception of the group that had not been acid etched at the second and third sequences, in which significantly less adhesive remnant was observed at the third sequence than at the first and second. These results may be explained by the fact that not etching does not create new porosity in the enamel, so reducing the microretention of the adhesive.

For the SEM observation, after the first sequence, the etching effects produced by the different procedures were similar, although the NRC showed a less aggressive etching pattern. However, at the second and third sequence, composite remnants were observed in all groups despite the fact that the enamel surfaces had been cleaned with a finishing carbide bur until all visible remnants had been eliminated. These observations agree with those of Bishara *et al.* (2003), who used the acid-etch technique for all bond/rebond sequences. The presence of these remnants may explain the gradual reduction in bond strength for each successive sequence within each group, given that these remnants contribute to a reduction in the roughness of the enamel and to the appearance of porosity when the tooth surface is repeatedly conditioned (Bishara *et al.*, 2003).

In group 2, in which acid etching was omitted at the second and third sequence, a greater reduction in surface roughness was noted at the third sequence than at the first and second, which, together with the cohesion characteristics of the material itself after polymerization, may explain the significant decrease in bond strength.

It must be taken into account that *in vitro* studies have their limitations. However, they are necessary and useful for initial evaluation of adhesive systems. However, *in vivo* research must be carried out to confirm *in vitro* results.

Conclusions

Both NRC and TSEP achieve a bond comparable with the acid-etch technique when new brackets are bonded repeatedly up to three times on the same tooth. When the acid-etch technique is used to condition enamel, etching need not be repeated when a bracket is bonded to a tooth for a second time but should be repeated for subsequent bonding.

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