European Journal of Orthodontics 28 (2006) 173–178
doi:10.1093/ejo/cji089
Advance Access publication 20 January 2006

Self-etching primer and a non-rinse conditioner versus phosphoric acid: alternative methods for bonding brackets

Ascensión Vicente*, Luis A. Bravo* and Martín Romero**
Units of *Orthodontics and **Pediatric Dentistry, Dental Clinic, University of Murcia, Spain

SUMMARY The objective of this study was to evaluate the effectiveness of a self-etching primer, Transbond Plus Self Etching Primer (TSEP, 3M Unitek), and a non-rinse conditioner (NRC, Dentsply DeTrey) for bonding brackets, compared with the acid-etch technique. The brackets were bonded to extracted premolars using Transbond XT (3M Unitek). One of the following three conditioning protocols were used: (1) 37 per cent phosphoric acid (n = 25), (2) TSEP (n = 25), and (3) NRC (n = 15). Shear bond strength (SBS) was measured with a universal testing machine. The adhesive remaining after debonding was determined using image analysis equipment. Scanning electron microscope (SEM) observations were also carried out on 12 premolars to observe the enamel surfaces.

No significant differences were observed in SBS between the three groups ($P = 0.56$). TSEP and NRC left significantly less adhesive on the tooth than the traditional acid-etch technique ($P = 0.004$ and $P = 0.000$, respectively). NRC left significantly less adhesive than TSEP ($P = 0.001$). SEM observations showed that NRC produced a less aggressive etch pattern than TSEP, and that the etching effect of TSEP approximated that of phosphoric acid.

Introduction

Phosphoric acid and its derivatives were used for treating metallic surfaces in order to adhere paints, resins, etc. Based on this fact, Buonocore (1955) found an increase in the adhesion of acrylic materials used at that time when the enamel surface had been previously treated with an 85 per cent phosphoric acid solution for 30 seconds. This procedure resulted in one of the most important advances in dentistry, improving the nature of resin–enamel bonds and generating new clinical applications such as pit and fissure sealants, resin restorations, adhesion of orthodontic brackets, bridges, etc. (Hasshimoto *et al.*, 2003)

It is estimated that the enamel surface lost during etching prior to the bonding of brackets is between 10 and 30 µm. On the other hand, removal of adhesive remnants on the enamel surface after debonding results in a reduction of enamel of approximately 55.6 µm (Bishara *et al.*, 2000). One of the main research objectives in orthodontics concerning adhesion, is to improve the bonding procedure by minimizing the loss of enamel during the bonding and debonding of brackets without compromising the bond strength (Bishara *et al.*, 2001a).

The effects of reducing the acid concentration (Wang *et al.*, 1994; Bhad and Hazarey, 1995; Cartensen, 1995) and the time of application (Sheen *et al.*, 1993; Osorio *et al.*, 1999; Gardner and Hobson, 2001) have been investigated, as well the use of alternative acids (Urabe *et al.*, 1999; Çehreli and Altay, 2000; Gardner and Hobson, 2001). Alternative methods to acid etching have also been proposed, such as air abrasion (Reisner *et al.*, 1997; Canay *et al.*, 2000; Van Waveren Hogerverst *et al.*, 2000) or laser etching (Von Fraunhofer *et al.*, 1993).

Alternatives to acid etching have recently been introduced in orthodontics in an attempt to reduce enamel loss and simplify bonding procedures. These methods include the use of self-etching primers and non-rinse conditioners (NRCs).

As the name suggests, an NRC etches the enamel without the need for rinsing. The literature concerning its use in orthodontics is limited. Çehreli and Altay (2000) observed that its application produced a smooth yet 'adequately rough' enamel surface for bonding. However, Bishara *et al.* (2001a) reported unacceptable bond strengths when combining an NRC with a compomer.

Self-etching primers combine etching and priming simultaneously; thus simplifying application. There is controversy concerning the use of self-etching primers to etch enamel. Some investigations show that they provide bond strengths comparable with those obtained with the acid-etch technique (Arnold *et al.*, 2002; Cacciafesta *et al.*, 2003; Dorminey *et al.*, 2003) whilst others have observed significantly lower bond strengths (Bishara *et al.* 2001b, 2002; Yamada *et al.*, 2002; Zeppieri *et al.*, 2003).

The objective of this study was to evaluate the bonding effectiveness of a self-etching primer, Transbond Plus Self Etching Primer (TSEP, 3M Unitek Dental Products, Monrovia, California, USA), and an NRC (Dentsply DeTrey, Konstanz, Germany) in comparison with the traditional acid-etch technique. Scanning electron microscope (SEM) observations were also carried out to observe the enamel surfaces treated with each product.

Materials and methods

Teeth

Seventy-seven sound human maxillary premolars were used. These had been extracted for reasons unrelated to the objectives of this study and with the informed consent of the patients. The project was approved by the Murcia University Bio-ethical Commission.

The teeth were washed in water to remove any traces of blood and then placed in 0.1 per cent Timol solution. They were then stored in distilled water which was changed periodically to avoid deterioration. In no case was a tooth stored for more than 1 month after extraction.

For shear bond strength (SBS) testing, 65 premolars were used. The teeth were set in a 4 cm long copper cylinder with an internal diameter of 3 cm and their roots set in type IV plaster.

For SEM observations, 12 premolars were used.

Brackets

Sixty-five metal maxillary premolar brackets were used (Victory Series, 3M Unitek). The base area of each bracket was calculated (mean = 9.79 mm^2) using image analysis equipment and MIP 4 software (Microm Image Processing Software, Digital Image Systems, Barcelona, Spain).

Bonding procedure

The maxillary premolars were divided into three groups and the brackets were bonded buccally. The manufacturers' instructions were followed throughout the study.

For all groups the buccal surfaces were polished with a rubber cup and a glycerine-free polishing paste (Détartrine, Septodont, Saint-Maur, France).

Group I (n = 25): Acid + Transbond XT primer + Transbond XT paste (3M Unitek). The teeth were etched with 37 per cent *o*-phosphoric acid gel (Total Etch, Ivoclar, Vivadent. Schaan, Liechtenstein) for 30 seconds, and then thoroughly washed and air dried. A layer of Transbond XT primer was applied to the tooth and Transbond XT paste applied to the base of the bracket which was then placed on the tooth with firm pressure. Excess adhesive was removed from around the base of the bracket and the adhesive was then light-cured, positioning the light guide of an Ortholux XT lamp (3M Unitek) on each interproximal side for 10 seconds.

Group II (n = 25): TSEP + Transbond XT paste. The enamel was treated with TSEP, which was gently rubbed onto the enamel for 3 seconds. A moisture-free air source was used to deliver a gentle burst of air to the enamel and the bracket bonded with Transbond XT paste as in group I.

Group III (n = 15): NRC + Transbond XT primer + Transbond XT paste. NRC was gently brushed onto the enamel leaving it undisturbed for 20 seconds. A moisture-free air source was used to deliver a gentle burst of air to the enamel and the bracket bonded with Transbond XT (primer and paste) as in group I.

Storage of test specimens

The specimens were immersed in distilled water at a temperature of 37°C for 24 hours (International Organization for Standardization, 1994).

Bond strength test

SBS was measured with a universal test machine (Autograph AGS-1KND, Shimadzu, 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 (International Organization for Standardization, 1994).

The teeth were set at the base of the machine so that the sharp end of the rod contacted the area between the base and the wings of the bracket, exerting a force parallel to the tooth surface in an occluso-gingival direction (Figure 1).

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

Adhesive Remnant Index

The percentage of the surface of the bracket base covered by adhesive was determined using image analysis equipment (Sony dxc 151-ap video camera, connected to an Olympus

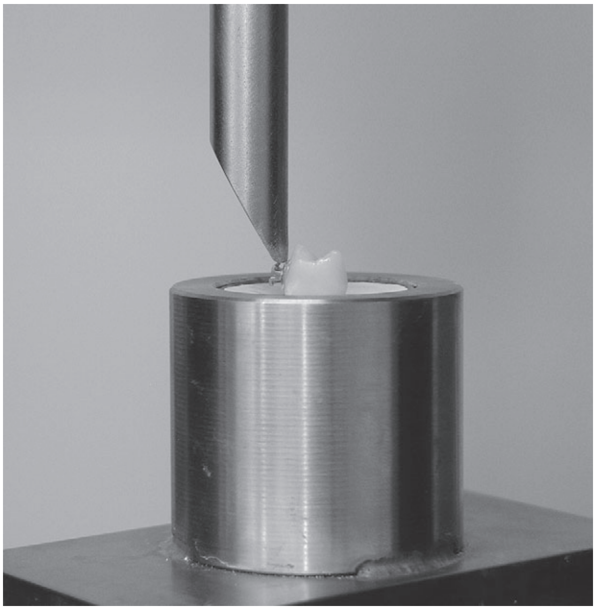

Figure 1 Shear bond strength test.

SZ11 microscope (Digital Image Systems) and MIP 4 software (Digital Image Systems).

The estimation of the error of the method of the adhesive remnant quantification, obtained with the image analysis equipment was carried out as follows: the adhesive remaining on 25 brackets was measured with the image analysis equipment twice with a period of 6 months between the measurements. The results of the two measurements were compared with a *t*-test for two dependent samples. No significant differences were found ($P < 0.05$).

The percentage of the area with adhesive remaining on the tooth after debonding was obtained by subtracting the area of adhesive covering the bracket base from 100 per cent. Each tooth was then assigned an Adhesive Remnant Index (ARI) value according to the following criteria (Årtun and Bergland, 1984):

0 = No adhesive left on the tooth.
1 = Less than half of the adhesive left on the tooth.
2 = More than half of the adhesive left on the tooth.
3 = All the adhesive left on the tooth.

Possible enamel fractures were also registered macroscopically.

Statistical analysis

The Kolmogorov–Smirnov normality test, the Shapiro–Wilk normality test, and Levene's variance homogeneity test were applied to the bond strength data. As the data did not show a normal distribution, significant differences were evaluated using the Kruskal–Wallis test ($P < 0.05$).

Bond strength data were analysed with Kaplan–Meier survival analysis using the Breslow statistic ($P < 0.05$).

ARI values were analysed with Pearson's chi-square test. An analysis of corrected residuals was also carried out to detect particular associations for the different groups and ARI scores. Both statistical tests were repeated grouping the cases in categories with scores 0 and 1 or scores 2 and 3, with the aim of avoiding categories showing an expected frequency lower than 5. A significance level of $P < 0.05$ was set for both Pearson's chi-square test and the analysis of corrected residuals (residual > 2 implies $P < 0.05$).

The Kolmogorov–Smirnov, the Shapiro–Wilk test, and Levene's homogeneity test of variances were applied to the data for percentage of area of adhesive remaining on tooth. As there was neither homogeneity of variance nor a normal distribution, they were analysed using the Kruskal–Wallis test. To determine those groups which were significantly different, the Mann–Whitney test for two independent samples was used. In order to avoid an accumulation of errors due to multiple comparisons (to compare the three groups two by two, three comparisons were made) a Bonferroni correction was carried out. The significance level ($P < 0.05$) was modified by dividing by the number of comparisons made, and therefore $P < 0.017$ was considered significant.

SEM observation

SEM was used to observe the effect of conditioning with phosphoric acid, NRC, and TSEP on the buccal enamel surface. Twelve premolars were divided into three groups. The crowns were sectioned from the roots with a diamond disc at the cementolabial enamel junction, and each crown was cut longitudinally in a mesiodistal direction. Group 1: the enamel was polished with a rubber cup and polishing paste, etched with 37 per cent *o*-phosphoric acid gel for 30 seconds, followed by rinsing and drying. Group 2: the enamel was polished with a rubber cup and polishing paste, treated with TSEP and rinsed with acetone for 10 seconds to remove the self-etching primer (Kanemura *et al.*, 1999). Group 3: the enamel was polished with a rubber cup and polishing paste, and NRC was applied to the enamel leaving it undisturbed for 20 seconds. A moisture-free air source was used to deliver a gentle burst of air to the enamel.

All specimens were cleaned in distilled water with ultrasonic agitation for 30 minutes and gently air dried. They were then fixed to SEM stubs, coated with gold and examined under a 6100 SEM (Joel Limited, Tokyo, Japan) operating at 15 kV. Representative images for the different surface treatments were captured digitally and stored in computer files.

Results

The results for bond strength are shown in Table 1. The Kruskal–Wallis test did not show significant differences ($P = 0.56$) between the groups (Table 1), nor did the Kaplan–Meier survival analysis detect significant differences in bond strength for the different groups ($P = 0.53$; Figure 2).

Pearson's chi-square test showed significant differences ($P < 0.05$) for the ARI. The analysis of corrected residuals demonstrated that while the acid etching group was significantly associated (residue = 3.3) with ARI 2, NRC was associated with ARI 1 (residue = 3.6). TSEP was not significantly associated with any particular ARI (Table 2).

Both tests were repeated grouping the values obtained for scores 0 and 1, and scores 2 and 3. Significant differences ($P < 0.05$) were also obtained when the chi-square test was applied, and a significant association was found for the acid etching group (residue = 3.3) in the '2 + 3' category, and for NRC in the '0 + 1' category (residue = 3.5). TSEP showed no significant associations (Table 3).

The percentage of tooth area occupied by adhesive remnants is shown in Table 4. Kruskal–Wallis test detected significant differences. The Mann–Whitney testing for two independent samples showed that these differences were between the acid etch group and NRC ($P = 0.000$), the acid etch group and TSEP ($P = 0.004$), and TSEP and NRC ($P = 0.001$).

Figure 3 shows the SEM observations. Treatment with NRC resulted in fine surface roughening and pitted enamel surfaces. The enamel treated with phosphoric acid showed

Table 1 Shear bond strengths (MPa).

Group	*n*	Mean	Median	Standard deviation	Minimum	Maximum
Phosphoric acid	25	12.27	11.30	5.01	6.79	28.01
Transbond Plus Self Etching Primer	25	12.20	11.34	4.27	6.13	23.10
Non-rinse conditioner	15	10.45	12.05	4.09	4.65	15.60

Kruskal–Wallis test did not show significant differences between groups ($P = 0.56$).

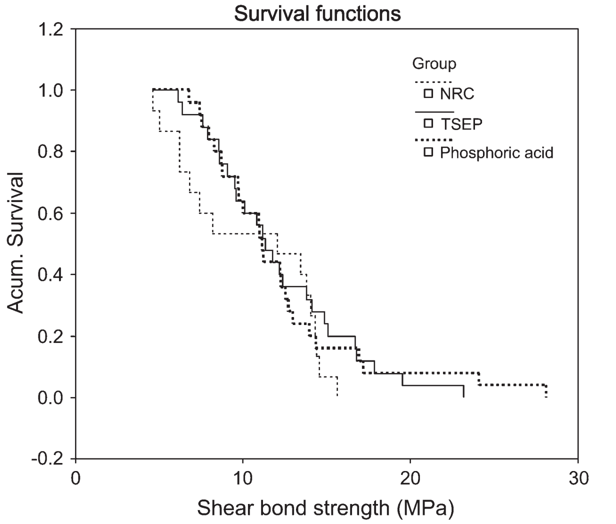

Figure 2 Probability of failure of the different groups at particular shear stress values. $P = 0.53$.

Table 2 Adhesive Remnant Index (ARI).

Group	*n*	ARI			
		0	1	2	3
Phosphoric acid	25	0	7	18[a]	0
Transbond Plus Self Etching Primer	25	1	13	11	0
Non-rinse conditioner	15	0	14[a]	1	0

ARI values were analysed by means of Pearson's chi-square test and an analysis of corrected residuals.
[a]indicates the ARI value to which each group was significantly associated ($P < 0.05$).

Table 3 Adhesive Remnant Index (ARI) values grouped into categories.

Group	*n*	ARI	
		0 + 1	2 + 3
Phosphoric acid	25	7	18[a]
Transbond Plus Self Etching Primer	25	14	11
Non-rinse conditioner	15	14[a]	1

ARI values were grouped and evaluated using Pearson's chi-square test (obtaining significant differences) and an analysis of corrected residuals.
[a]indicates the class to which each group was significantly associated ($P < 0.05$).

a rougher surface, and an overall increase in microporosity was evident. The enamel treated with TSEP produced a porous surface, with the etching effect approximating that with use of phosphoric acid.

Discussion

The results showed no significant differences in bond strength between the three groups and are in agreement with some *in vitro* investigations, which demonstrated comparable bond strengths when using TSEP and phosphoric acid as the conditioners (Arnold *et al.*, 2002; Cacciafesta *et al.*, 2003; Dorminey *et al.*, 2003; Larmour and Stirrups, 2003). However, Zeppieri *et al.* (2003) observed that TSEP resulted in a significantly lower bond strength than the acid-etch technique. Conversely, Buyukyilmaz *et al.* (2003) found that the use of TSEP provided significantly greater bond strength than etching the enamel with phosphoric acid. Ireland *et al.* (2003), in an *in vivo* study, stated that the percentage of bracket failure was greater using TSEP than the acid-etch technique.

Studies concerning the use of NRC for bonding brackets are limited. Bishara *et al.* (2001a) reported unacceptable bond strengths when using NRC together with a compomer. In an investigation on ground enamel, Sunico *et al.* (2002) observed no significant differences between etching the enamel with 36 per cent phosphoric acid for 30 seconds and using a NRC to adhere a composite resin.

Significant differences were observed in the ARI between the three groups. TSEP and NRC left significantly less adhesive on the tooth than the traditional acid-etch technique, and NRC left significantly less adhesive than TSEP. This is clinically advantageous as adhesive removal implies loss of enamel (Bishara *et al.*, 2000) and less chairside time is required when removing adhesive.

These results are in agreement with others showing that TSEP left less adhesive on the tooth than when the acid-etching technique was used (Cacciafesta *et al.*, 2003; Larmour and Stirrups, 2003). However, other investigators

Table 4 Percentage of tooth area occupied by adhesive.

Group	n	Mean	Median	Standard deviation	Minimum	Maximum
Phosphoric acid[a]	25	59.88	60.82	19.20	17	89
Transbond Plus Self Etching Primer[b]	25	39.80	25.89	25.00	0	79
Non-rinse conditioner[c]	15	15.90	11.42	14.00	3	56

The results were evaluated using the Kruskal–Wallis test and the Mann–Whitney test for two independent samples. Groups marked with different superscript letters showed significant differences with one another ($P < 0.017$).

have found that TSEP left adhesive on the tooth that did not differ from that remaining when the enamel was conditioned with phosphoric acid (Buyukyilmaz *et al.*, 2003; Zeppieri *et al.*, 2003).

SEM observations showed that NRC produced a modest etch pattern when compared with TSEP, and that the etching effect of TSEP approximated that of phosphoric acid. These results agree with those of Pashley and Tay (2001) who observed that NRC produced a less aggressive etch pattern than a self-etching primer used in restorative dentistry, and furthermore that the etching effect of this self-etching primer was similar to the effect of phosphoric acid. NRC produced a less aggressive etch pattern than phosphoric acid and TSEP. Despite this, the etch pattern was sufficiently retentive for orthodontic bonding; its bond strength was comparable with TSEP and phosphoric acid.

a b c

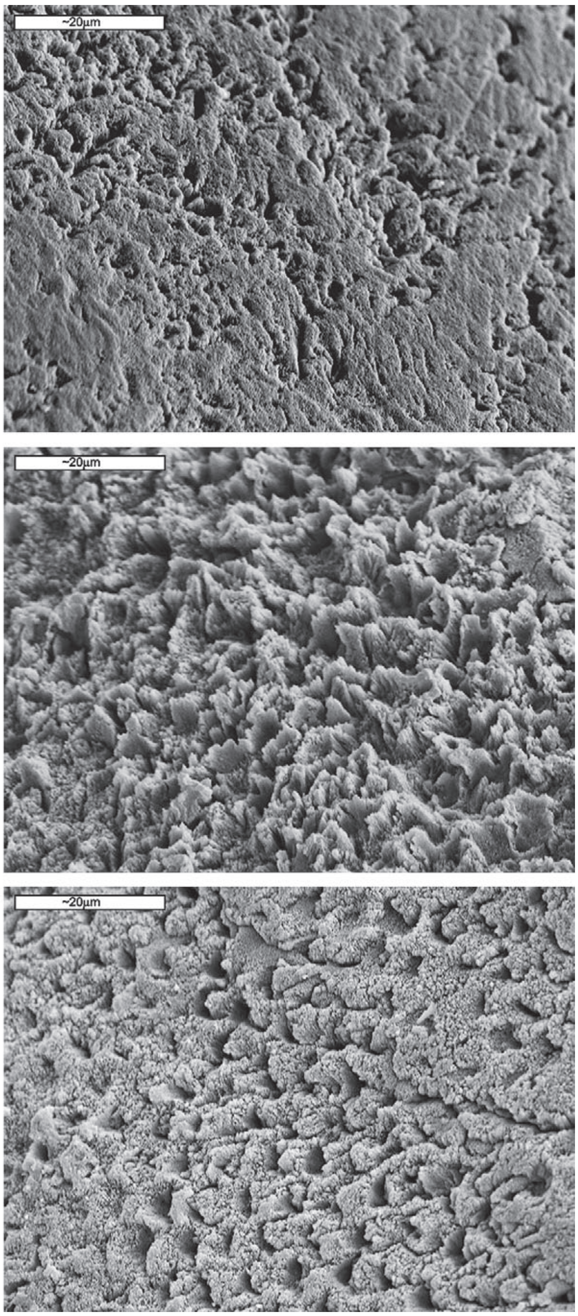

Figure 3 Scanning electron micrographs of enamel after the application of (a) non-rinse conditioner, (b) 37 per cent phosphoric acid for 30 seconds, and (c) Transbond Plus Self Etching Primer.

Conclusion

Both TSEP and NRC are adequate for bonding. However, caution must be exercised when extrapolating laboratory results to the clinical situation as there is a complex relationship between *in vitro* bond strength mean values and adhesive failures *in vivo* (Pickett *et al.*, 2001). Therefore, *in vivo* research must follow *in vitro* investigations.

Address for correspondence

Dr A. Vicente,
Unit of Orthodontics
Dental Clinic
University of Murcia
Hospital Morales Meseguer
2ª planta
C/Marqués de los Vélez, s/n
30008 Murcia
Spain
E-mail: ascenvi@um.es

References

Arnold R W, Combe E C, Warford Jr J H 2002 Bonding of stainless steel brackets to enamel with a new self-etching primer. American Journal of Orthodontics and Dentofacial Orthopedics 122: 274–276

Årtun J, Bergland S 1984 Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. American Journal of Orthodontics 85: 333–340

Bhad W A, Hazarey P V 1995 Scanning electron microscopic study and shear bond strength measurements with 5% and 37% phosphoric acid. American Journal of Orthodontics and Dentofacial Orthopedics 108: 410–414

Bishara S E, VonWald L, Laffoon J F, Jackobsen J R 2000 Effect of altering the type of enamel conditioner on the shear bond strength of a resin-reinforced glass ionomer adhesive. American Journal of Orthodontics and Dentofacial Orthopedics 118: 288–294

Bishara S E, Laffoon J F, VonWald L, Warren J J 2001a Evaluation of nonrinse conditioning solution and a compomer as an alternative method of bonding orthodontic brackets. Angle Orthodontist 71: 461–465

Bishara S E, VonWald L, Laffoon J F, Warren J J 2001b Effect of a self-etch primer/adhesive on the shear bond strength of orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 119: 621–624

Bishara S E, Ajlouni R, Laffoon J F, Warren J J 2002 Effect of a fluoride-releasing self-etch acidic primer on the shear bond strength of orthodontic brackets. Angle Orthodontist 72: 199–202

Buonocore M G 1955 A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. Journal of Dental Research 34: 849–853

Buyukyilmaz T, Usumez S, Karaman A I 2003 Effect of self-etching primers on bond strength – are they reliable? Angle Orthodontist 73: 64–70

Cacciafesta V, Sfondrini M F, De Angelis M, Scribante A, Klersy C 2003 Effect of water and saliva contamination on shear bond strength of brackets bonded with conventional, hydrophilic, and self-etching primers. American Journal of Orthodontics and Dentofacial Orthopedics 123: 633–640

Canay S, Kocadereli I, Akça E 2000 The effect of enamel air abrasion on the retention of bonded metallic orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 117: 15–19

Cartensen W 1995 Effect of reduction of phosphoric acid concentration on the shear bond strength of brackets. American Journal of Orthodontics and Dentofacial Orthopedics 108: 274–277

Çehreli Z C, Altay I 2000 Effects of a nonrinse conditioner and 17% ethylenediaminetetraacetic acid on the etch pattern of intact human permanent enamel. Angle Orthodontist 71: 22–27

Dorminey J C, Dunn W J, Taloumis L J 2003 Shear bond strength of orthodontic brackets bonded with a modified 1-step etchant-and-primer technique. American Journal of Orthodontics and Dentofacial Orthopedics 124: 410–413

Gardner A, Hobson R 2001 Variations in acid-etch patterns with different acid and etch times. American Journal of Orthodontics and Dentofacial Orthopedics 120: 64–67

Hasshimoto M, Ohno H, Yoshida E, Hoiri M, Kaga M, Oguchi H 2003 Resin-enamel bonds made with self-etching primers on ground enamel. European Journal of Oral Science 111: 447–453

International Organization for Standardization 1994 Dental materials – guidance on testing of adhesion to tooth structure. Geneva, Switzerland, ISO TR 11405

Ireland A J, Knight H H, Sherriff M 2003 An *in vivo* investigation into bond failure rates with a new self-etching primer system. American Journal of Orthodontics and Dentofacial Orthopedics 124: 323–326

Kanemura N, Sano H, Tagami J 1999 Tensile bond strength to and SEM evaluation of ground and intact enamel surfaces. Journal of Dentistry 27: 523–530

Larmour C J, Stirrups D R 2003 An *ex vivo* assessment of a bonding technique using a self-etching primer. Journal of Orthodontics 30: 225–228

Osorio R, Toledano M, García-Godoy F 1999 Bracket bonding with 15- or 60-second etching and adhesive remaining on enamel after debonding. Angle Orthodontist 69: 45–49

Pashley D H, Tay F R 2001 Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel. Dental Materials 17: 430–444

Pickett K L, Sadowsky P L, Jacobson A, Lacefield W 2001 Orthodontic *in vivo* bond strength: comparison with *in vitro* results. Angle Orthodontist 71: 141–148

Reisner K R, Levitt H L, Mante F 1997 Enamel preparation for orthodontic bonding: a comparison between the use of a sandblaster and current techniques. American Journal of Orthodontics and Dentofacial Orthopedics 111: 366–373

Sheen D H, Wang W N, Tarng T H 1993 Bond strength of younger and older permanent teeth with various etching times. Angle Orthodontist 63: 225–230

Sunico M C, Shinkai K, Medina III V O, Shirono M, Tanaka N, Katoh Y 2002 Effect of surface conditioning and restorative material on the shear bond strength and resin-dentin interface of a new bottle nanofilled adhesive. Dental Materials 18: 535–542

Urabe H, Rossouw P E, Titley K C, Yamin C 1999 Combinations of etchants, composite resins, and bracket systems: an important choice in orthodontic bonding procedures. Angle Orthodontist 69: 267–275

Van Waveren Hogervers W L, Feilzer A J, Prahl-Andersen B 2000 The air-abrasion technique versus the conventional acid-etching technique: a quantification of surface enamel loss and comparison of shear bond strength. American Journal of Orthodontics and Dentofacial Orthopedics 117: 20–26

Von Fraunhofer J A, Allen D J, Orbell G M 1993 Laser etching of enamel for direct bonding. Angle Orthodontist 63: 73–76

Wang W N, Yeh C L, Fang B D, Sun K T, Arvystas M G 1994 Effect of H_3PO_4 concentration on bond strength. Angle Orthodontist 64: 377–382

Yamada R, Hayakawa T, Kasai K 2002 Effect of using self-etching primer for bonding orthodontic brackets. Angle Orthodontist 72: 558–564

Zeppieri I L, Chung C-H, Mante F K 2003 Effect of saliva on shear bond strength of an orthodontic adhesive used with moisture-insensitive and self-etching primers. American Journal of Orthodontics and Dentofacial Orthopedics 124: 414–419