European Journal of Orthodontics 31 (2009) 271–275
doi:10.1093/ejo/cjn103
Advance Access publication 20 February 2009

Comparison of a recently developed nanofiller self-etching primer adhesive with other self-etching primers and conventional acid etching

Güvenç Başaran, Törün Özer and Jalan Devecioğlu Kama
Department of Orthodontics, Faculty of Dentistry, Dicle University, Diyarbakır, Turkey

SUMMARY The purpose of this *ex vivo* study was to compare the bond strengths and modes of failure of brackets bonded with three self-etching products [Adper Prompt L-Pop (3M Espe), Futurabond NR (Voco) and Transbond Plus (3M Unitek)] and a conventional 38 per cent phosphoric acid-etching system Etch-Rite (Pulpdent Corp.) at two different time points. Ninety-six defect-free premolars were randomly allocated to eight groups ($n=12$). The etching procedure was carried out according to the manufacturers' instructions. The shear bond strengths (SBSs) were measured after storage in distilled water for 12 and 24 hours. The brackets were debonded using a universal testing machine with a cross-head speed of 0.5 mm/minute. Multiple comparisons of the SBSs for the different etching types were performed using analysis of variance. The chi-square test was used to evaluate differences in adhesive remnant index (CARI) scores among the groups.

The differences between SBSs analysed at 12 and 24 hours were not significantly different. Although a significant difference ($P < 0.01$) was observed with conventional and self-etching primer (SEP) adhesive systems, SEP revealed bond strengths higher than clinically accepted limits (6–8 MPa). SEP systems can achieve successful orthodontic bond strengths. The rate of development of the bond strength must be determined to ensure sufficient maturation of orthodontic adhesives before functional loading.

Introduction

Since the report by Buonocore (1955), the standard protocol to remove the smear layer for successful bonding has been acid etching. Although phosphoric acid etching is a reliable technique in orthodontics, there is a need to simplify clinical procedures and minimize enamel loss while maintaining a clinically useful bond strength (Powers *et al.*, 1997; Mandall *et al.*, 2002). The irregular enamel surface created by dissolving hydroxyapatite crystals permits penetration of the fluid adhesive components, and this penetration provides micromechanical retention. Acid etching of enamel appears to improve retention by selectively eroding specific hydroxyapatite formations and facilitating penetration by the development of resin tags (Silverstone *et al.*, 1975; Sakaki *et al.*, 1994). Development of these micromechanical bonds contributes to long-term bonding strength.

Advances in adhesive technology have led orthodontists to incorporate new adhesives, composite resins, and bonding techniques into clinical practice. Self-etching primer (SEP) products, which combine acid and primer, simplify the bonding procedure, reduce chair time, and avoid the side-effects of acid etching (Büyükyilmaz *et al.*, 2003; Grubisa *et al.*, 2004; Sirirungrojying *et al.*, 2004).

The efficacy of SEPs regarding adhesion to enamel has been studied; although clinically acceptable, lower values for shear bond strength (SBS) were reported when compared with conventional multistep etch and prime systems (Silverstone *et al.*, 1975; Mandall *et al.*, 2002).

In a clinical situation, bond failure can occur soon after the orthodontic bracket is positioned as a result of stress (Katona, 1997; Knox *et al.*, 2001). This could be caused by the orthodontic procedure, contraction of the adhesive, or normal oral functions such as mastication. Sufficient bond strength is a factor that contributes to the clinical success of orthodontic treatment, and data collected after 24 hours have generally been used to measure the bond strengths of orthodontic adhesives (Fox *et al.*, 1994; Stanford *et al.*, 1997). Previous reports have highlighted the importance of the early bond strength of orthodontic adhesives and shown that materials exposed to the oral environment must be strong enough to withstand both short- and long-term forces (Whitlock *et al.*, 1994; Mitchell *et al.*, 1995; Trites *et al.*, 2004). Some previous studies focused on the immediate bond strength achieved by adhesive systems, by measurement at selected time intervals over a 24 hour period (Bin Abdullah and Rock, 1996; Chamda and Stein, 1996).

Recent advances in bonding systems have lead to the use of nanotechnology in dentistry. Futurabond NR (Voco, Cuxhaven, Germany) is a new dentine–enamel bond reinforced with nanofillers. The manufacturer claims that this self-etching adhesive is suitable for all light-curing restorative procedures.

This *ex vivo* study aimed to determine and compare the SBSs and mode of failure of brackets bonded with (1) a recently developed modification of the self-etching adhesive system; (2) a fluoride-releasing, self-etching adhesive system; (3) a newly developed nano-reinforced self-etching adhesive system; and (4) a conventional 38 per cent phosphoric acid etching system. A further aim was to determine if there was a significant difference in SBSs between these four etching systems related to time by comparing them at 12 and 24 hours.

Materials and methods

A total of 96 human premolars were used in this study. After extraction, the teeth were stored in 0.9 per cent NaCl solution at 4°C. The criteria for tooth selection included intact buccal enamel with no cracks caused by the extraction forceps and no caries. After extraction, all enamel surfaces were examined under a light stereomicroscope (S240, Olympus, Tokyo, Japan) at ×10 magnification. The teeth were assigned to eight groups each of 12 specimens using random number tables (Figure 1). The teeth were cleansed of soft tissue, embedded in cold-curing, fast-setting acrylic (Leocryl, Leone, Sesto Fiorentino, Italy), and placed in a plastic box. Each tooth was orientated so that its labial surface was parallel to the applied force during shear bond testing.

The orthodontic etching systems used in this study were Adper Prompt L-Pop self-etch adhesive (3M Espe, St Paul, Minnesota, USA), nano-reinforced Futurabond NR single dose (Voco), Transbond Plus SEP (3M Unitek, Monrovia, California, USA), and Etch-Rite (Pulpdent Corp., Watertown, Massachusetts, USA) 38 per cent phosphoric acid. All orthodontic etching systems were used according to the manufacturers' instructions. For the phosphoric acid group, the original primer of the Transbond XT adhesive was used.

After etching, stainless steel standard edgewise premolar brackets (GAC, Central Islip, New York, USA) were bonded to the teeth with a surface bonding area of 12.6 mm^2. A thin, uniform coating of adhesive agent was applied to the etched surface. After application of Transbond XT, the bracket was placed onto the tooth surface, adjusted to its final position, and pressed firmly into place. Excess sealant and adhesive were removed from the periphery of the bracket base with a probe to ensure a uniform bonding area. Light curing was performed for a total of 40 seconds by irradiating the mesial, distal, occlusal, and gingival aspects of the tooth for 10 seconds each.

Another important point in testing the *ex vivo* SBS of adhesives is the light consistency and intensity. The device (Hilux LED Max, Benlioglu Dental Inc., Ankara, Turkey) used in the study had an internal light metre and between each curing episode, the device was tested for intensity (Figure 2).

After bonding, the specimens were transferred to distilled water at 37°C and stored for 12 and 24 hours. SBS testing was accomplished with a chisel edge mounted on the cross-head of a testing machine (Instron Testometric M500–25 KN, Testometric Company Ltd, Rochdale, Lancashire, UK). The chisel edge contacted at the bracket and enamel interface at a crosshead speed of 0.5 mm/minute; the force decay was recorded in Newtons and then converted to megapascals by dividing the force by the bracket base area. All tests were conducted at 24 ± 2°C.

After debonding, the brackets were examined under ×10 magnification to evaluate the amount of resin remaining on the tooth. The adhesive remnant index (ARI) was used to describe the quantity of resin remaining on the tooth surface (Årtun and Bergland, 1984).

Descriptive statistics, including the mean, standard deviation, and minimum and maximum values, were calculated for each group. Multiple comparisons of SBSs for the different etching types were performed using analysis of variance. The chi-square test was used to evaluate differences in ARI scores

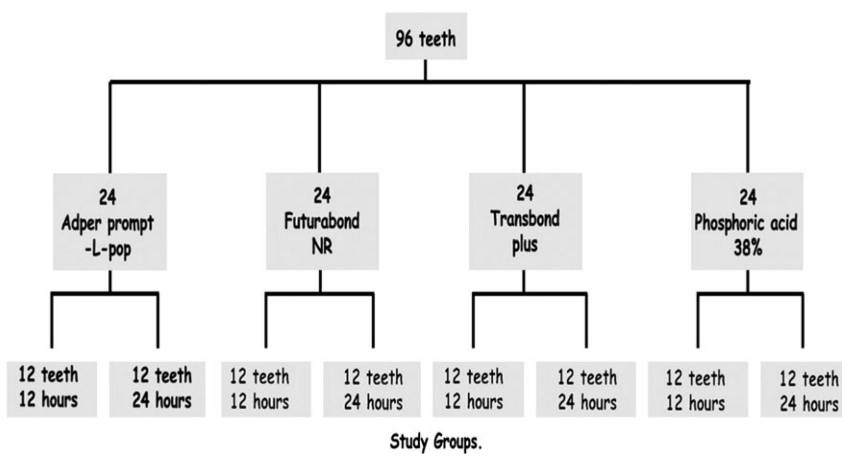

Figure 1 Flow chart of allocation to the groups.

Figure 2 Light device used in the study.

among the groups. All statistical evaluations were made using the Statistical Package for Social Sciences (Windows, release 10.0.0, SPSS Inc., Chicago, Illinois, USA).

Results

Shear bond strengths

Descriptive statistics for the comparison of SBS and ARI scores for the four groups are given in Tables 1–3. Table 1 shows the observed SBS at 12 and 24 hours. Although SBS at 24 hours was generally higher than that observed at 12 hours, there was no statistically significant difference at either time point. The mean SBS achieved with the conventional acid etch system was statistically significantly greater ($P < 0.01$) than that observed with the SEP adhesive systems. There was no statistically significant difference in bond strength between the SEP adhesive systems.

Because there was no statistical significance of time on SBS, the 12 and 24 hour groups were combined (Table 2). Again, there was no statistically significant difference between the SEP adhesive systems but a significant difference was observed when compared with the conventional acid-etching system ($P < 0.01$).

ARI scores

The ARI scores (Table 3) indicate the site of bond failure for the acid- and self-etched groups at the 12 and 24 hour debonding times. The scores were not statistically different from each other when time and adhesive system were considered as the variables. A slight tendency towards a score of 3 was noted with debonding at 24 hours but this was not statistically significant. Similarly, ARI scores achieved with conventional acid-etching systems had a slight tendency towards a score of 3, indicating that more adhesive was left on the enamel surface.

Discussion

Although clinical orthodontic procedures have improved, there is still a need to enhance adhesive systems to shorten bonding procedures and eliminate enamel loss without jeopardizing clinical performance. This has led manufacturers to develop SEP adhesive systems, to reduce the steps required, and to decrease chair-side time and technique sensitivity (Büyükyilmaz *et al.*, 2003; Grubisa *et al.*, 2004; Sirirungrojying *et al.*, 2004).

In general, SEPs are considered as bicomponent hydrophilic adhesives containing hydrofluoric complexes, deionized water, stabilizer in one compartment and activator, orthophosphoric ester methyacrylate, and stabilizers in a separate compartment (Bishara *et al.*, 2002; Velo *et al.*, 2002). The acidic monomer becomes neutralized when it is applied to the enamel surface. The pH is raised and demineralization occurs as a result of recruited calcium ions from the hydroxyapatite crystals. The encapsulated ions in the primer provide infiltration of the adhesive for micromechanical adhesion of the resin (Bishara *et al.*, 1999; Brosnihan and Safranek, 2000; Bishara *et al.*, 2002; Velo *et al.*, 2002; Büyükyilmaz *et al.*, 2003; Grubisa *et al.*, 2004). According to the manufacturer of Futurabond NR, nanoparticles acting as cross links reinforce the bond and hybrid layer. The manufacturer claims that the minute size of the nanofillers allows complete penetration which contributes to high adhesion. However, this was not supported by the results of this study. If the manufacturers' claims were valid, the SBS of the nanofilled material would have been higher than that of the other adhesives; yet the observed mean SBS of Futurabond NR (7.9 ± 2.5 MPa) was the lowest of all four adhesives, although there was no statistically significant difference among the SEP adhesive systems. Fillers are added to the adhesives to provide strengthening, increase stiffness, and reduce dimensional changes (Ferracane *et al.*, 1987; Kim *et al.*, 1994). Previous studies (Miyazaki *et al.*, 1991; James *et al.*, 2003; Faltermeier *et al.*, 2007) demonstrated that the filler content influences polymerization shrinkage. In the light of these findings, it is suggested that smaller filler particles lead to greater shrinkage which results in low bond strengths, which was observed with Futurabond NR.

Table 1 Shear bond strengths in megapascals of the etchants used in the study measured at 12 and 24 hours [A, Adper Prompt L-Pop; B, Transbond Plus; C, Futurabond NR (single dose); D, 38% phosphoric acid].

	Etching systems	Mean (MPa)	Standard deviation	Standard error	Maximum	Minimum	Significance
12 hours	A	8.1	2.4	1.2	14.9	6.3	NS
	B	9.2	2.4	0.9	17.6	6.4	NS
	C	7.4	2.6	1.1	18.4	4.9	NS
	D	13.2	1.9	1.0	23.2	9.1	**
24 hours	A	8.4	2.5	1.1	15.1	6.6	NS
	B	10.2	2.8	1.3	19.0	5.6	NS
	C	8.6	2.6	1.3	17.7	6.2	NS
	D	15.6	2.6	1.1	24.5	9.4	*

NS, not significant. $^{*}P < 0.05$, $^{**}P < 0.01$.

Table 2 Comparison of the shear bond strengths in megapascals of the four etchants used in the study [A, Adper Prompt L-Pop; B, Transbond Plus; C, Futurabond NR (single dose); D, 38% phosphoric acid].

Etching systems	Mean (MPa)	Standard deviation	Standard error	Maximum	Minimum	Significance
A	8.3	2.4	1.2	15.1	6.3	NS
B	9.7	2.6	1.2	19.0	5.6	NS
C	7.9	2.5	1.2	18.4	4.9	NS
D	14.4	2.2	1.0	24.5	9.1	**

NS, not significant. $^{**}P < 0.01$.

Table 3 Adhesive remnant index scores at 12 and 24 hours for the four etchants used in the study [A, Adper Prompt L-Pop; B, Transbond Plus; C, Futurabond NR (single dose); D, 38% phosphoric acid].

	12 hours				24 hours				Chi-square test
	0	1	2	3	0	1	2	3	
A	2	5	5	0	2	5	2	3	4.217, $P > 0.05$
B	1	4	7	0	1	5	5	1	
C	2	4	6	0	0	5	6	1	
D	1	4	5	1	0	4	6	2	

0, no adhesive remained on the tooth; 1, less than half of the enamel-bonding site covered with adhesive; 2, more than half of the enamel-bonding site covered with adhesive; and 3, the enamel-bonding site covered entirely with adhesive.

All SEP adhesive systems (Adper Prompt L-Pop, 8.3 ± 2.4 MPa; Transbond Plus, 9.7 ± 2.6 MPa; Futurabond NR, 7.9 ± 2.5 MPa) had statistically significantly lower mean SBS values than the conventional acid etch system (14.4 ± 2.2 MPa). Reynolds (1975) reported that bond strengths of 6–8 MPa were clinically acceptable. The observed mean SBSs with the SEP adhesive systems were approximately consistent with the upper limit of this range and were similar to those found by Arnold *et al.* (2002).

The reason for using SEP adhesives (Adper Prompt L-Pop and Transbond Plus) in this study was not to justify their suitability for orthodontic bonding since this recommendation is evident from previous studies (Bishara *et al.*, 1999; Brosnihan and Safranek, 2000; Velo *et al.*, 2002; Büyükyilmaz *et al.*, 2003; Grubisa *et al.*, 2004; Sirirungrojying *et al.*, 2004) but to compare the recently developed nanofiller with other SEP adhesives in addition to a conventional acid-etching adhesive system.

Adequate bond strength is a factor that contributes to the clinical success of orthodontic treatment. Data collected after 24 hours have generally been used to measure the bond strengths of orthodontic adhesives (Fox *et al.*, 1994; Yamamoto *et al.*, 2006). The earliest time point at which there was no significant difference in bond strength compared with that at 24 hours was defined as the initial stable time. In the present study, the aim was to determine if a stable bond strength was reached at 12 hours or if it continued to increase up to 24 hours. Although there was no statistically significant difference between bond strengths at either time point, generally the bond strengths at 24 hours tended to be higher. Adper Prompt L-Pop showed values of 8.1 ± 2.4 and 8.4 ± 2.5 MPa, Transbond Plus 9.2 ± 2.4 and 10.2 ± 2.8 MPa, and Futurabond NR 7.4 ± 2.6 and 8.6 ± 2.6 MPa at 12 and 24 hours, respectively. Conventional acid etching revealed a higher mean bond strength than all SEP adhesives at 12 (13.2 ± 1.9 MPa) and 24 (15.6 ± 2.6 MPa) hours.

There was no statistically significant difference in ARI scores at 12 or 24 hours. All adhesives showed higher scores with debonding at 24 hours, i.e. more adhesive was left on the enamel surface. This could be considered to be either an advantage or a disadvantage. While less chair time is needed with less adhesive left on enamel after debonding, there is an inverse risk of enamel fracture while debonding.

Conclusion

All adhesives tested achieved clinically acceptable bond strengths. Although an adequate bond strength is reached at 12 hours, all adhesives continue strengthen up to 24 hours.

Address for correspondence

Güvenç Başaran
Department of Orthodontics
Faculty of Dentistry
Dicle University
21280 Diyarbakır
Turkey
E-mail: basaran@dicle.edu.tr

References

Arnold R W, Combe E C, Warford 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

Bin Abdullah M, Rock W P 1996 The effect of etch time and debond interval upon the shear bond strength of metallic orthodontic brackets. British Journal of Orthodontics 23: 121–124

Bishara S E, Gordon V V, VonWald L, Jakobsen J R 1999 Shear bond strength of composite, glass ionomer and acidic primer adhesive systems. American Journal of Orthodontics and Dentofacial Orthopedics 115: 24–28

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

Brosnihan J, Safranek L 2000 Orthodontic bonding: the next generation. Journal of Clinical Orthodontics 10: 614–616

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

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

Chamda R A, Stein E 1996 Time-related bond strengths of light-cured and chemically cured bonding systems: an *in vitro* study. American Journal of Orthodontics and Dentofacial Orthopedics 110: 378–382

Faltermeier A, Rosentritt M, Faltermeier R, Reicheneder C, Müβig D 2007 Influence of filler level on the bond strength of orthodontic adhesives. Angle Orthodontist 77: 494–498

Ferracane J L, Antonio R C, Matsumoto H 1987 Variables affecting the fracture toughness of dental composites. Journal of Dental Research 66: 1140–1145

Fox N A, McCabe J F, Buckley J G 1994 A critique of bond strength testing in orthodontics. British Journal of Orthodontics 21: 33–43

Grubisa H S, Heo G, Raboud D, Glover K E, Major P W 2004 An evaluation and comparison of orthodontic bracket bond strengths achieved with self-etching primer. American Journal of Orthodontics and Dentofacial Orthopedics 126: 213–219

James J W, Miller B H, English J D, Tadlock L P, Buschang P H 2003 Effects of high-speed curing devices on shear bond strength and microleakage of orthodontic brackets. American Journal of Orthodontics and Dentofacial Orthopedics 123: 555–561

Katona T R 1997 Stresses developed during clinical debonding of stainless steel orthodontic brackets. Angle Orthodontist 67: 39–46

Kim K H, Park J H, Imai Y, Kishi T 1994 Microfracture mechanisms of dental resin composites containing spherically-shaped filler particles. Journal of Dental Research 73: 499–504

Knox J, Kralj B, Hübsch P F, Middleton J, Jones M L 2001 An evaluation of the influence of orthodontic adhesive on the stresses generated in a bonded bracket finite element model. American Journal of Orthodontics and Dentofacial Orthopedics 119: 43–53

Mandall N A, Millett D T, Mattick C R, Hickman J, Worthington H V, Macfarlane T V 2002 Orthodontic adhesives: a systematic review. Journal of Orthodontics 29: 205–210

Mitchell C A, O'Hagan E, Walker J M 1995 Probability of failure of orthodontic brackets bonded with different cementing agents. Dental Materials 11: 317–322

Miyazaki M, Hinoura K, Onose H, Moore B K 1991 Effect of filler content of light-cured composites on bond strength to bovine dentine. Journal of Dentistry 19: 301–303

Powers J M, Kim H B, Turner D S 1997 Orthodontic adhesives and bond strength testing. Seminars in Orthodontics 3: 147–156

Reynolds I R 1975 A review of direct orthodontic bonding. British Journal of Orthodontics 2: 171–178

Sakaki T, Fukushima T, Kawai S, Matsumoto M 1994 Effect of physical properties of direct bonding adhesives on bonding to etched enamel. Journal of Prosthetic Dentistry 71: 552–559

Silverstone L M, Saxton C A, Dogan I L, Fejerskov O 1975 Variation in the pattern of acid etching on human dental enamel examined by scanning electron microscopy. Caries Research 9: 373–387

Sirirungrojying S, Saito K, Hayakawa T, Kasai K 2004 Efficacy of using self-etching primer with a 4-META/MMA-TBB resin cement in bonding orthodontic brackets to human enamel and effect of saliva contamination on shear bond strength. Angle Orthodontist 74: 251–258

Stanford S K, Wozniak W T, Fan P L 1997 The need for standardization of test protocols. Seminars in Orthodontics 3: 206–209

Trites B, Foley T F, Banting D 2004 Bond strength comparison of 2 self-etching primers over a 3-month storage period. American Journal of Orthodontics and Dentofacial Orthopedics 126: 709–716

Velo S, Carano A, Carano A 2002 Self-etching vs. traditional bonding systems in orthodontics: an *in vitro* study. Orthodontics and Craniofacial Research 5: 166–169

Whitlock 3rd B O, Eick J D, Ackerman R J, Glaros A G, Chappell R P 1994 Shear strength of ceramic brackets bonded to porcelain. American Journal of Orthodontics and Dentofacial Orthopedics 106: 358–364

Yamamoto A, Yoshida T, Tsubota K, Takamizawa T, Kurokawa H, Miyazaki M 2006 Orthodontic bracket bonding: enamel bond strength vs time. American Journal of Orthodontics and Dentofacial Orthopedics 130: 435–441