4-1-2012

Bonding with Self-etching Primers – Pumice or Pre-etch? An *in vitro* Study

Ian J. Fitzgerald
Marquette University

Gerard T. Bradley
Marquette University

Jose A. Bosio
Marquette University, jose.bosio@marquette.edu

Arthur F. Hefti
Marquette University, arthur.hefti@marquette.edu

David W. Berzins
Marquette University, david.berzins@marquette.edu

Accepted version. *European Journal of Orthodontics*, Vol. 34, No. 2 (April 2012): 257-261. DOI.

European Journal of Orthodontics. Vol. 34. No. 2 (April, 2012): 257-261. DOI.

Contents

Bonding with self-etching primers—pumice or pre-etch? An *in vitro* study

Ian Fitzgerald
Department of Developmental Studies, Marquette University of Dentistry, Milwaukee, WI
Gerard T. Bradley
Department of Developmental Studies, Marquette University of Dentistry, Milwaukee, WI
Jose A. Bosio
Department of Developmental Studies, Marquette University of Dentistry, Milwaukee, WI
Arthur F. Hefti
Department of Developmental Studies, Marquette University of Dentistry, Milwaukee, WI
David W. Berzins
Department of Developmental Studies, Marquette University of Dentistry, Milwaukee, WI

Abstract

The purpose of this study was to compare the shear bond strengths (SBSs) of orthodontic brackets bonded with self-etching primer (SEP) using different enamel surface preparations. A two-by-two factorial study design was used. Sixty human premolars were harvested, cleaned, and randomly assigned to four groups (n = 15 per group). Teeth were bathed in saliva for 48 hours to form a pellicle. Treatments were assigned as follows: group 1 was pumiced for 10 seconds and pre-etched for 5 seconds with 37 per cent phosphoric acid before bonding with SEP (Transbond Plus). Group 2 was pumiced for 10 seconds before bonding. Group 3 was pre-etched for 5 seconds before bonding. Group 4 had no mechanical or chemical preparation before bonding. All teeth were stored in distilled water for 24 hours at 37°C before debonding.

The SBS values and adhesive remnant index (ARI) score were recorded. The SBS values (±1 SD) for groups 1–4 were 22.9 ± 6.6, 16.1 ± 7.3, 36.2 ± 8.2, and 13.1 ± 10.1 MPa, respectively. Two-way analysis of variance and subsequent contrasts showed statistically significant differences among treatment groups. ARI scores indicated the majority of adhesive remained on the bracket for all four groups. Pre-etching the bonding surface for 5 seconds with 37 per cent phosphoric acid, instead of pumicing, when using SEPs to bond orthodontic brackets, resulted in greater SBSs.

Introduction

The orthodontic profession is constantly seeking to improve and optimize the technique of bonding brackets to enamel. Self-etching primers (SEPs) have been extensively researched (Barry, 1995; Bishara *et al.*, 2001; Pandis and Eliades, 2005; Burgess *et al.*, 2006; dos Santos *et al.*, 2006; Murfitt *et al.*, 2006; Davari *et al.*, 2007; Lill *et al.*, 2008) and have emerged as a successful alternative to the conventional acid-etch bonding technique. Since the introduction of SEPs, it has become accepted that pumicing the bonding surface beforehand to remove the salivary pellicle results in increased bond strength and decreased clinical failure rates (Burgess *et al.*, 2006; Lill *et al.*, 2008). A key to successful orthodontic bonding is removal of the salivary pellicle. In the conventional multi-step acid-etch bonding procedure, the pellicle is removed by application of 37 per cent phosphoric acid for 15–60 seconds; therefore, pumicing is not necessary (Barry, 1995; Lindauer *et al.*, 1997; Ireland and

Sherriff, 2002). Although marketed as reducing the number of steps in bonding by combining the conditioning and priming stages, the need for initial pumicing is reintroduced when using SEPs.

Concerns regarding the use of pumice include the time required to individually pumice each tooth and rinse away the paste, the possible introduction of gingival crevicular fluid proteins onto the enamel surface, and the potential for mechanical injury to the gingiva. However, elimination of pumicing from the SEP bonding sequence leaves a compromising salivary pellicle on the enamel. An alternative to pumicing to remove the pellicle when using SEPs would be to introduce an etching step. Anecdotal reports suggest a short 5–10 second pre-etch with 37 per cent phosphoric acid can result in a clinically superior performance when compared with pumicing, but no evidence exists in the literature to confirm the clinical effectiveness of the procedure. *In vitro* studies (Erhardt *et al.*, 2004; Lührs *et al.*, 2008) have shown consistently greater bond strengths when enamel was pretreated with phosphoric acid before bonding with SEPs. However, the teeth in these studies were not pumiced when bonding with SEPs.

The authors are not aware of any published studies that compared bond strengths between acid pretreated and pumiced enamel with the use of SEPs. Although some clinicians have adopted a pre-etch step in place of pumicing in their SEP bonding protocols, conclusive *in vitro* and *in vivo*studies examining this practice are needed. The aim of this *in vitro* study was to investigate shear bond strength (SBS) values of brackets bonded with an SEP to salivary pellicle-coated human teeth that were pretreated with pumice and/or 37 per cent phosphoric acid or not pretreated at all.

Materials and methods

Following approval from the Institutional Review Board at Marguette University, 60 human premolars were collected. The teeth were washed in running water, placed in distilled water, and stored at room temperature. Teeth chosen for the study were free of cracks, caries, and restorations.

A two-by-two factorial study design was used. Presence or absence of pumicing (P±) and pre-etching (E±) were the investigated effects, resulting in four treatment groups: group 1 (P+/E+), group 2 (P+/E−), group 3 (P−/E+), and group 4 (P−/E−). Group 2 follows the manufacturer recommendations for bonding with SEPs and thus could be considered a control group. Sixty teeth were selected and randomly assigned to treatments in blocks of four. The roots of all premolars were then removed and the teeth were vigorously scrubbed on their bonding surfaces with a toothbrush and running water to ensure a clean surface. Whole saliva was collected from the first author in a glass beaker. Cleaned teeth were immersed in saliva for 48 hours at 37°C on a shaking platform to form a pellicle on the enamel surfaces. Immediately before bonding, each tooth was individually removed from the saliva with tweezers and dried with oil-free compressed air until the surface appeared dry

Experimental group preparation and bonding

The first author performed all bonding procedures. The teeth allocated to P+/E+ were prepared by pumicing each tooth for 10 seconds with a rubber prophylactic cup and fluoride- and oil-free coarse pumice powder (Whip-Mix Corp, Louisville, Kentucky, USA) mixed with water, rinsing with distilled water, and drying with oil-free compressed air. Phosphoric acid (37 per cent) gel (3M Unitek, Monrovia, California, USA) was placed on the bonding surface of each tooth for 5 seconds, and the tooth was again rinsed and dried. Bonding orthodontic brackets was executed as per the manufacturer’s instructions. Transbond Plus self-etching primer (3M Unitek) was applied to the surface of each tooth and rubbed for 5 seconds. Next, the bonding surface received a gentle 5 second air burst. Adhesive pre-coated stainless steel universal bicuspid brackets (APC II™ Adhesive Coated Appliance System Victory Series™; 3M Unitek) with a 0.022 inch slot, 0 degrees of tip, 0 degrees of torque, and a 10 mm^2 base were placed on each tooth. The excess adhesive was removed with a fine probe. Each specimen was light cured (Ortholux LED Curing Light; 3M Unitek) for 10 seconds from the mesial and distal.

The other teeth were treated using protocols that included the following modifications: Teeth allocated to P+/E− were not pre-etched, P−/E+ were not pumiced, and P−/E− were neither pumiced nor pre-etched.

Debonding and classification of adhesive remnant index

Following bonding the brackets, each tooth was individually mounted in acrylic resin (Great Lakes Orthodontics, Tonawanda, New York, USA) using a consistent orientation. Next, they were stored in distilled water for 24 hours at 37°C. Each mounted tooth was then placed in a universal testing machine (Instron Corporation, Canton, Massachusetts, USA) with the bracket/tooth interface placed parallel to the blade motion. The blade (24 mm wide tapered to an edge 0.3 mm thick) moved from the occlusal direction and made contact evenly across the bracket as it contacted the space between the tie wings and bracket base as close to the base as possible. It should be noted that, although commonly reported as shear testing, a significant amount of peeling/normal forces are applied with this type of test arrangement (Katona, 1997). The brackets were debonded using a crosshead speed of 0.1 mm/minute. After debonding, each tooth and debonded bracket were viewed under an optical stereomicroscope at ×10 magnification. The adhesive remnant index (ARI) score (Artun and Bergland, 1984) was recorded to determine where the bond failure occurred. Possible ARI scores are 0 for no adhesive left on the tooth, 1 for less than half of the adhesive left on the tooth, 2 for more than half of the adhesive left on the tooth, and 3 for all the adhesive left on the tooth.

Statistical analysis

Descriptive statistics for each test group included means and standard deviations for SBS data and frequencies for ARI. SBSs were analysed using a two-way analysis of variance with 'pumicing' and 'pre-etching' as main factors. A total sample size of $N = 60$ was required to detect an effect size (ES) = 0.4, assuming equal treatment group sizes, a type I error probability $\alpha = 0.05$, and power $(1-\beta) = 0.85$ (Faul *et al.*, 2007). A Weibull analysis was performed to determine the Weibull modulus, characteristic strength, and bond strengths at specific reliabilities. The ARI scores were fit to a multinomial logistic regression model to determine whether pumicing or pre-etching before bonding was significant in predicting the ARI score. Statistical software (SPSS Inc., Chicago, Illinois, USA and SAS Institute Inc., Cary, North Carolina, USA) was used for computations.

Results

Detailed results of SBS measurements and their derivatives are presented in Table 1. Statistically significant effects were observed for both main factors as well as for their interaction. For that reason, the statistical analysis was continued by applying contrasts on interaction effects. As expected, the absence of any surface preparation (P−/E−) resulted in the lowest SBS values. In contrast, pre-etching alone (P−/E+) was the most effective preparation step. It was statistically different from P−/E− ($P < 0.0001$), P+/E− ($P < 0.0001$), and P+/E+ ($P < 0.0001$). The combination of pumicing and pre-etching was more effective than P−/E− ($P = 0.003$). There was no statistical difference between P+/E+ and P+/E−. The Weibull analysis (Table 1) shows P−/E+ presented with the greatest Weibull modulus, characteristic strength, and bond strengths at 10 and 90 per cent probability of failure, while P−/E− was the lowest in each.

Table 1 Shear bond strengths (SBSs) and Weibull analysis results.

Group	**Mean ± SD (MPa)**	**Weibull modulus (*β*)**	**Characteristic strength (*α*)**	**SBS (MPa) at 10% probability of failure**	**SBS (MPa) at 90% probability of failure**
1	22.9 ± 6.6[b]	3.6	25.1	13.5	31.6
2	16.1 ± 7.3[bc]	1.9	18.7	5.8	28.8
3	36.2 ± 8.2[a]	6.5	39.6	28.0	45.0
4	13.1 ± 10.0[c]	1.3	14.3	2.6	27.1

Different letters denote significant ($P < 0.05$) differences exist.

The ARI scores are presented in Table 2. Eighty-five per cent of the scores were either 0 or 1, indicating that after debonding, most adhesive remained on the bracket. Neither pumicing (P = 0.66) nor pre-etching (P = 0.91) was found to be statistically significant in predicting the ARI score. Six instances of enamel fractures were identified of which four were found in P+/E+ and two in P−/E+.

Table 2 Adhesive remnant index (ARI) scores by group. EF, enamel fracture.

Group	ARI scores*				EF
	0	1	2	3	
1	4	5	2	0	4
2	4	11	0	0	0
3	4	8	0	1	2
4	11	4	0	0	0

*

Neither pumicing (P = 0.66) nor pre-etching (P = 0.91) was found to be statistically significant in predicting the ARI score.

Discussion

The effects of two types of enamel surface preparations, pumicing, and pre-etching, on the SBS induced by a SEP were evaluated in this study. Pre-etching without pumicing when using SEPs produced greater mean bond strengths compared to that of the manufacturer's recommendation of pumicing before SEP application. It should be noted that the SBS values for P−/E+ were very high, although they are similar to values found in a few other orthodontic bonding studies (Theodorakopoulou *et al.*, 2004; Uysal *et al.*, 2010). While commonly performed, comparison of bond strength values across studies is problematic due to differences in methodological and testing parameters. Therefore, intra-study group comparisons are most valid. With this in mind, based upon this *in vitro*bonding study, pre-etching has been shown to be a possible alternative to pumicing when using SEPs.

Considering that the P+/E+ group was pumiced and pre-etched before bonding with the SEP, it could have been expected to have the greatest bond strengths or values similar to P−/E+. However, this was not confirmed by the results of this study. A possibility is that pumicing, pre-etching, and etching from the SEP may ‘over prepare’ the enamel surface, similar in concept to studies that showed beyond an optimal conventional etching time, bond strengths remain the same, or may actually decrease (Legler *et al.*, 1989; Wang and Lu, 1991; Reisner *et al.*, 1997). Alternatively, despite rinsing, pumice may have remained on the tooth and affected bond strength. Nevertheless, exposing enamel to both a pumicing and a short acid-etch pretreatment when bonding with SEPs is not a routine clinical protocol and would unlikely be adopted since only pumicing (P+/E−) or pre-etching (P−/E+) protocols provide clinically acceptable bond strength (Tavas and Watts, 1984), are simpler, and less time-consuming.

An added variable in this study was establishing a salivary pellicle on the enamel surface. Few, if any, SEP bonding studies have considered the effects of a salivary pellicle. Turk *et al.* (2007) examined whether saliva contamination affects the bond strength of SEPs, by brushing saliva across the prepared bonding surface, but the short saliva exposure times may not have been sufficient to form a pellicle. It has been shown by Hannig (1999) that an initial 10–20 nm layer of pellicle forms after 3 minutes of saliva immersion. After 2 hours, it varies between 80–200 nm (Hannig, 1999; Hannig *et al.*, 2001). In the current study, when no attempt was made to remove the salivary pellicle through pre-etching or pumicing, bonding effectiveness appeared compromised as evidenced by the low bond strength for P−/E−. Clinical trials (Burgess *et al.*, 2006; Lill *et al.*, 2008) have confirmed the importance of pumicing in the removal of the salivary pellicle before bonding with SEPs *in vivo*.

A majority of ARI scores for all four groups were 0 or 1, indicating that adhesive was more likely to remain on the bracket as opposed to the tooth after debonding. Clinically, this is desirable as it would require less time for clean-up of the enamel. Pre-coated brackets have been shown to leave less adhesive on the tooth compared to when adhesive is applied to the bracket base at the time of bonding (Vicente and Bravo, 2007). Enamel fractures were observed among teeth that were pre-etched before SEP bonding. This observation may be cause for concern even if *in vitro* bond strengths are not directly reflective of *in vivo* bond strengths. Grubisa *et al.*(2004) reported enamel fractures in a SEP study but attributed this to tooth storage in formalin. In the current study, the teeth were mounted in acrylic, which releases heat due to its exothermic setting reaction. It is conceivable that the increased heat weakened the enamel by inducing microcracks that coalesced under the greater stress observed in groups 1 and 3. Nevertheless, Retief (1974) has reported enamel fracture at bond strengths of only 9.7 MPa, reflecting the wide variation seen in bonding studies.

For teeth weakened by large restorations or aged teeth with existing cracks, pre-etching during bonding with SEPs may not be recommended. On the other hand, possible indications for pre-etching with SEPs include rebonding brackets that have debonded during active treatment, bonding teeth in areas of increased occlusal forces (second molars), bonding to aprismatic or irregular enamel, and bonding to surgically exposed teeth.

The results showed more than adequate bond strengths using a 5 second pre-etch. However, this may not be practical if bonding more than a few teeth with this technique, as by the time etchant is removed from the first tooth, more than 5 seconds may have elapsed. Clinically, leaving the etchant on for 10 seconds may be more realistic but this could potentially lead to excessive bond strengths when using SEPs. The cost benefit ratio also should be considered. A clinical trial would be necessary to determine if the greater expense of SEP compared with conventional primer is of such advantage to use a pre-etch/SEP technique over a conventional bonding technique.

In vitro studies have indicated acceptable bond strengths when using SEPs with pumicing (Bishara *et al.*, 2001). Additionally, clinical studies have shown relatively few debonds (Burgess *et al.*, 2006; Lill *et al.*, 2008). Nevertheless, anecdotal accounts mention that some clinicians are substituting a pumicing step with a pre-etching step during their SEP bonding protocol. Little evidence exists in the literature examining the effectiveness of this practice. The results obtained in this study suggest that pre-etching enamel prior to SEP application allows absolute exposure of the enamel to the SEP, fully removing the salivary pellicle, maximizing primer penetration of the enamel, and therefore maximizing bond strength. Clinical studies examining debond and enamel fracture rates are needed before fully endorsing this procedure.

Conclusions

Within the limitations of this *in vitro* bonding study, the results showed

1. A 5 second pre-etch with 37 per cent phosphoric acid, when bonding with SEPs, gives significantly greater bond strengths compared with pumicing.
2. The majority of adhesive remained on the bracket for all four groups with no significant difference observed with pumicing or pre-etching.

Funding

This study was supported by a small grant to the first author from the Marquette University School of Dentistry Office of Research and Graduate Studies.

The authors wish to thank 3M Unitek for their generous donation of materials and Dr Jessica E. Pruszynski from the Medical College of Wisconsin for statistical consultation.

References

Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment, American Journal of Orthodontics , 1984, vol. 85 (pg. 333-340)

Barry GR. A clinical investigation of the effects of omission of pumice prophylaxis on band and bond failure, British Journal of Orthodontics , 1995, vol. 22 (pg. 245-248)

Bishara SE, VonWald L, Laffoon JF, Warren JJ. Effect of a self-etch primer/adhesive on the shear bond strength of orthodontic brackets, American Journal of Orthodontics and Dentofacial Orthopedics , 2001, vol. 119 (pg. 621-624)

Burgess AM, Sherriff M, Ireland AJ. Self-etching primers: is prophylactic pumicing necessary? A randomized clinical trial, Angle Orthodontist , 2006, vol. 76 (pg. 114-118)

Davari AR, Yassaei S, Daneshkazemi AR, Yosefi MH. Effect of different types of enamel conditioners on the bond strength of orthodontic brackets, Journal of Contemporary Dental Practice , 2007, vol. 8 (pg. 36-43)

dos Santos JE, Quioca J, Loguercio AD, Reis A. Six-month bracket survival with a self-etch adhesive, Angle Orthodontist , 2006, vol. 76 (pg. 863-868)

Erhardt MC, Cavalcante LM, Pimenta LA. Influence of phosphoric acid pretreatment on self-etching bond strengths, Journal of Esthetic and Restorative Dentistry , 2004, vol. 16 (pg. 33-40)

Faul F, Erdfelder E, Lang AG, Buchner A. G*Power 3: a flexible statistical power analysis program for the social, behavioral, and biomedical sciences, Behavior Research Methods , 2007, vol. 39 (pg. 175-191)

Grubisa HS, Heo G, Raboud D, Glover KE, Major PW. An evaluation and comparison of orthodontic bracket bond strengths achieved with self-etching primer, American Journal of Orthodontics and Dentofacial Orthopedics , 2004, vol. 126 (pg. 213-219)

Hannig M. Ultrastructural investigation of pellicle morphogenesis at two different intraoral sites during a 24-h period, Clinical Oral Investigations , 1999, vol. 3 (pg. 88-95)

Hannig M, Herzog S, Willigeroth SF, Zimehl R. Atomic force microscopy study of salivary pellicles formed on enamel and glass in vivo, Colloid and Polymer Science , 2001, vol. 279 (pg. 479-483)

Ireland AJ, Sherriff M. The effect of pumicing on the in vivo use of a resin modified glass poly(alkenoate) cement and a conventional no-mix composite for bonding orthodontic brackets, Journal of Orthodontics , 2002, vol. 29 (pg. 217-220)

Katona TR. A comparison of the stresses developed in tension, shear peel, and torsion strength testing of direct bonded orthodontic brackets, American Journal of Orthodontics and Dentofacial Orthopedics , 1997, vol. 112 (pg. 244-251)

Legler LR, Retief DH, Bradley EL, Denys FR, Sadowsky PL. Effects of phosphoric acid concentration and etch duration on the shear bond strength of an orthodontic bonding resin to enamel: an in vitro study, American Journal of Orthodontics and Dentofacial Orthopedics , 1989, vol. 96 (pg. 485-492)

Lill DJ, Lindauer SJ, Tüfekçi E, Shroff B. Importance of pumice prophylaxis for bonding with self-etch primer, American Journal of Orthodontics and Dentofacial Orthopedics , 2008, vol. 133 (pg. 423-426)

Lindauer SJ, Browning H, Shroff B, Marshall F, Anderson RH, Moon PC. Effect of pumice prophylaxis on the bond strength of orthodontic brackets, American Journal of Orthodontics and Dentofacial Orthopedics , 1997, vol. 111 (pg. 599-605)

Lührs AK, Guhr S, Schilke R, Borchers L, Guertsen W, Günay H. Shear bond strength of self-etch adhesives to enamel with additional phosphoric acid etching, Operative Dentistry , 2008, vol. 33 (pg. 155-162)

Murfitt PG, Quick AN, Swain MV, Herbison GP. A randomised clinical trial to investigate bond failure rates using a self-etching primer, European Journal of Orthodontics , 2006, vol. 28 (pg. 444-449)

Pandis N, Eliades T. A comparative in vivo assessment of the long-term failure rate of 2 self-etching primers, American Journal of Orthodontics and Dentofacial Orthopedics , 2005, vol. 128 (pg. 96-98)

Reisner KR, Levitt HL, Mante F. Enamel preparation for orthodontic bonding: a comparison between the use of a sandblaster and current techniques, American Journal of Orthodontics and Dentofacial Orthopedics , 1997, vol. 111 (pg. 366-373)

Retief DH. Failure at the dental adhesive-etched enamel interface, Journal of Oral Rehabilitation , 1974, vol. 1 (pg. 265-284)

Tavas MA, Watts DC. A visible light-activated direct bonding material: an in vitro comparative study, British Journal of Orthodontics , 1984, vol. 11 (pg. 33-37)

Theodorakopoulou LP, Sadowsky PL, Jacobson A, Lacefield WJr. Evaluation of the debonding characteristics of 2 ceramic brackets: an in vitro study, American Journal of Orthodontics and Dentofacial Orthopedics , 2004, vol. 125 (pg. 329-336)

Turk T, Elekdag-Turk S, Isci D, Cakmak F, Ozkalayci N. Saliva contamination effect on shear bond strength of self-etching primer with different debond times, Angle Orthodontist , 2007, vol. 77 (pg. 901-906)

Uysal T, Ustdal A, Kurt G. Evaluation of shear bond strength of metallic and ceramic brackets bonded to enamel prepared with self-etching primer, European Journal of Orthodontics , 2010, vol. 32 (pg. 214-218)

Vicente A, Bravo LA. Shear bond strength of precoated and uncoated brackets using a self-etching primer, Angle Orthodontist , 2007, vol. 77 (pg. 524-527)

Wang WN, Lu TC. Bond strength with various etching times on young permanent teeth, American Journal of Orthodontics and Dentofacial Orthopedics , 1991, vol. 100 (pg. 72-79)