Original Article

Effect of a Fluoride-Releasing Self-Etch Acidic Primer on the Shear Bond Strength of Orthodontic Brackets

Samir E. Bishara, BDS, DDS, DOrtho, MS[a]; Raed Ajlouni, BDS[b]; John F. Laffoon, BS[c]; John J. Warren, DDS, MS[d]

Abstract: Conventional adhesive systems use three different agents—an enamel conditioner, a primer solution, and an adhesive resin— during the bonding of orthodontic brackets to enamel. A unique characteristic of some new bonding systems in operative dentistry is that they combine the conditioning and priming agents into a single application. Combining conditioning and priming saves time and should be more cost-effective to the clinician and indirectly to the patient. The purpose of this study was to assess and compare the effects of self-etching primers, including a fluoride-releasing primer, on the shear bond strength of orthodontic brackets. The brackets were bonded to extracted human teeth according to one of four protocols. In group 1 (control), teeth were etched with 37% phosphoric acid; after the sealant was applied, the brackets were bonded with Transbond XT (3M Unitek, Monrovia, Calif) and light cured for 20 seconds. In group 2, a self-etch acidic primer (3M ESPE, St Paul, Minn) was applied as suggested by the manufacturer, and the brackets were then bonded with Transbond XT as in the first group. In group 3, an experimental self-etch primer EXL #547 (3M ESPE) was applied to the teeth as suggested by the manufacturer, and the brackets were then bonded as in groups 1 and 2. In group 4, a fluoride-releasing self-etch primer, One-Up Bond F (J. Mortia, USA Inc, Irvine, Calif) that also has a novel dye-sensitized photo polymerization initiator system was applied as suggested by the manufacturer, and the brackets were then bonded as in the other groups. The present in vitro findings indicated that the shear bond strengths of the four groups were significantly different ($P = .001$). Duncan multiple range tests indicated that One-Up Bond F (mean ± SD strength, 5.1 ± 2.5 MPa) and Prompt L-Pop (strength, 7.1 ± 4.4 MPa) had significantly lower shear bond strengths than both the EXL #547 self-etch primer (strength, 9.7 ± 3.7 MPa) or the phosphoric acid etch and the conventional adhesive system (strength, 10.4 ± 2.8 MPa). (*Angle Orthod* 2002;72:199–202.)

Key Words: Self-etch primers; Fluoride release; Shear bond strength; Orthodontic brackets

INTRODUCTION AND LITERATURE REVIEW

Conventional adhesive systems use three different agents—an enamel conditioner, a primer solution, and an adhesive resin—in the process of bonding orthodontic brackets to enamel. A unique characteristic of some new bonding systems in operative dentistry is that they combine the conditioning and priming agents into a single acidic primer solution for simultaneous use on both enamel and dentin.[1,2] Combining conditioning and priming agents into a single treatment step results in reduced time and improved cost-effectiveness for the clinician and indirectly for the patient.

These relatively new systems were used originally on dentin.[1,3] Essentially, the acidic part of the primer dissolves the smear layer and incorporates it into the mixture. Acidic primer solutions also demineralize the dentin and encapsulate the collagen fibers and hydroxyapatite crystals.[2] This simultaneous conditioning and priming allows penetration of the monomer into the dentin. The adhesive resin component will then diffuse into the primed dentin, producing a ''hybrid layer.''[3] These new systems were found to also be effective when bonding to enamel.[4]

In the early 1990s, maleic acid was introduced as an alternative etching material, in an attempt to control the depth of the enamel etch. Barkmeier et al[5] compared 10% maleic acid with 37% phosphoric acid and reported that the

[a] Professor, Orthodontic Department, College of Dentistry, University of Iowa, Iowa City, Iowa.

[b] Graduate Resident, Department of Operative Dentistry, College of Dentistry, University of Iowa, Iowa City, Iowa.

[c] Research Assistant, College of Dentistry, University of Iowa, Iowa City, Iowa.

[d] Assistant Professor, Department of Preventive and Community Dentistry, College of Dentistry, University of Iowa, Iowa City, Iowa.

Corresponding author: Samir E. Bishara, BDS, DDS, DOrtho, MS, Orthodontic Department, College of Dentistry, University of Iowa, Iowa City, IA 52242.
(e-mail: karla-stsarkovich@uiowa.edu).

Accepted: October 2001. Submitted: August 2001.

resulting bond strengths were essentially similar (38.0 ± 6.5 MPa and 38.3 ± 8.0 MPa, respectively). Scanning electron microscopy of the enamel surfaces treated with 10% maleic acid and 37% phosphoric acid revealed a similar morphologic pattern, but the depth of the etched surface was significantly less with maleic acid.[6]

Orthodontists use the acid-etch bonding technique as a means of attaching brackets to the enamel surface. Maintaining a sound unblemished enamel surface after debonding orthodontic brackets is a primary concern to the clinician. As a result, bond failure at the bracket-adhesive interface or within the adhesive is more desirable (safer) than at the adhesive-enamel interface, since enamel fracture and crazing have been reported at the time of bracket debonding, especially with ceramic brackets.[7] As a result, alternative enamel conditioners, such as maleic acid and acidic primers, have been tested to determine if they can attain a clinically useful orthodontic bracket bond strength while decreasing the depth of enamel dissolution and decreasing the number of steps during the bonding procedure.[8]

Prompt (ESPE Dental AG, Seefeld, Germany) was introduced as an all-in-one adhesive for composites and compomers. The material can be light cured separately or after the application of the cavity restoration or the orthodontic adhesive. Prompt contains methacrylated phosphoric acid esters that combine an acidic component for etching the enamel and the primer. In a recent study,[9] the use of this self-etch primer provided significantly lower (but clinically useful) shear bond strength when compared with the use of phosphoric acid and a sealant before bonding orthodontic brackets with a composite adhesive.

In another study (SE Bishara et al, in preparation), an experimental self-etch primer (EXL #547, 3M ESPE, St Paul, Minn) was evaluated and was found to provide similar shear bond strengths to those obtained with the conventional adhesive systems.

Recently, a new self-etch primer, One-Up Bond F (J. Morita, USA Inc, Irvine, Calif) that contains and releases fluoride ions has been introduced.[10] The product also has a novel dye-sensitized photopolymerization initiator system.[10,11] If One-Up Bond F provides adequate shear bond strength, the fluoride release is an added advantage to minimize decalcification around orthodontic brackets.

The purpose of this study was to assess and compare the shear bond strength of three self-etch primers when used to bond orthodontic brackets.

MATERIALS AND METHODS

Teeth

Eighty freshly extracted human molars were collected and stored in a solution of 0.1% (weight/volume) thymol. The criteria for tooth selection included intact buccal enamel, the absence of any pretreatment with chemical agents (eg, hydrogen peroxide), the absence of cracks due to the presence of the extraction forceps, and the absence of caries. The teeth were cleansed and then polished with pumice and rubber prophylactic cups for 10 seconds.

Brackets used

Orthodontic central incisor metal brackets (Victory Series, 3M Unitek, Monrovia, Calif) were used in this study. The average bracket base surface area was 11.9 mm.[2]

Bonding procedure

The brackets were bonded to the teeth according to one of four protocols according to the manufacturers' instructions.

Group 1 (control). Twenty teeth were etched with 37% phosphoric acid gel for 30 seconds. The teeth were thoroughly washed and dried. The sealant was applied, and the brackets were bonded with Transbond XT (3M Unitek) and light cured for 20 seconds according to the manufacturer's instructions.

Group 2. On 20 teeth, the self-etch primer Prompt L-Pop (3M ESPE) containing both the acid and the primer was placed on the enamel for 15 seconds and gently evaporated with air, according to the manufacturer's instructions. The material used in the present study is predosed so that it is only used for one application. The unidose system in Prompt L-Pop has two compartments: one contains methacrylated phosphoric acid esters, initiators, and stabilizers, while the other contains water, fluoride complex, and stabilizers. For activation, the two compartments are squeezed into each other, and the resulting mix can be applied directly on the tooth surface. The brackets were then bonded with Transbond XT and light cured for 20 seconds as in Group 1.

Group 3. On 20 teeth, the enamel surface was lightly dried. The EXL #547 (3M ESPE) is made of two components, and a drop of each was mixed thoroughly for 5–10 seconds and rubbed on the enamel surface for 10 seconds. This layer was left to stand for 20 seconds, and the surface was lightly air dried and cured for 10 seconds. The brackets were then bonded with Transbond XT and cured for 20 seconds as in Groups 1 and 2. The self-etch primer EXL #547 contains organophosphorous compounds, methacrylate resin, 2 hydroxethyl-methacrylate (HEMA), polyalkenoic acid copolymer, ethanol, water, and a photo initiator.

Group 4. On 20 teeth, One-Up Bond F (J. Morita, USA Inc) was applied. According to the manufacturer, One-Up Bond F is designed to release fluoride for at least two years.[10] One-Up Bond F is provided as two solutions. Agent A contains phosphoric monomer, Propanedioic acid, [10-[(2-methyl-1-oxo-2-propenyl)oxy]decyl]-(9CI) (MAC-10) (adhesive promoting), bisphenol-A-glycidyl Methacrylate (Bis-GMA), multifunctional methacrylic monomers, and co-initiator. Agent B contains HEMA, monofunctional monomers, water, silicate filler, dye-sensitizer, and borate

TABLE 1. Descriptive Statistics and Results of the Analysis of Variance and Duncan Multiple Range Test Comparing the Shear Bond Strengths of the Four Groups[a]

Groups	Mean	SD	Range	Duncan[b]
Acid + primer + Transbond XT	10.4	2.8	6.4–19.1	A
Prompt L-Pop (self-etch primer) + Transbond XT	7.1	4.4	1.0–18.6	B
EXL# 547 (experimental self-etch primer) + Transbond XT	9.7	3.7	4.2–16.3	A
One-Up Bond F (fluoride-releasing self-etch primer) + Transbond XT	5.1	2.5	1.0–10.6	B

[a] Values are expressed in megapascals. F value = 10.21, $P = .001$ by analysis of variance.
[b] Groups with the same letter are not significantly different from each other.

derivative. When the two agents are mixed, the One-Up Bond F liquid turns from yellow to pink. The mixture is then applied to the tooth and left for 20 seconds before light curing for 15 seconds. At that point, the self-etch primer layer on the tooth surface should turn to a pale brown color confirming polymerization.[11] The brackets were bonded with Transbond XT adhesive as in the other groups. Each bracket was subjected to a 300-g compressive force with a force gauge (Correx Co, Bern, Switzerland) for 10 seconds, following which excess bonding resin was removed with a sharp scaler. All samples were stored in deionized water at 37°C for 48 hours.

Debonding procedure

The teeth were embedded in acrylic in phenolic rings (Buehler Ltd, Lake Bluff, Ill). A mounting jig was used to align the facial surface of the tooth perpendicular with the bottom of the mold. Each tooth was oriented with the testing device as a guide, so its labial surface was parallel to the force during the shear strength test. A steel rod with one flattened end was attached to the crosshead of a Zwick test machine (Zwick Gm bH & Co, Ulm, Germany). An occlusogingival load was applied to the bracket producing a shear force at the bracket-tooth interface. A computer, electronically connected with the Zwick test machine, recorded the results of each test. Shear bond strengths were measured at a crosshead speed of five mm/min.

Statistical analysis

Descriptive statistics including the mean, SD, and minimum and maximum values were calculated for each of the three test groups. Analysis of variance was used to determine if significant differences were present in the bond strength between the three groups. If significant differences were present, Duncan multiple range test was used to determine which of the means were significantly different from each other. Significance for all statistical tests was predetermined at $P \leq .05$.

RESULTS

The descriptive statistics for the shear bond strengths of the four groups evaluated are presented in Table 1. The results of the analysis of variance (F-value = 10.21) indicated that the shear bond strengths of the four groups were significantly different ($P = .001$). Duncan multiple range test indicated that One-Up Bond F (mean strength, 5.1 ± 2.5 MPa) and Prompt L-Pop (mean strength, 7.1 ± 4.4 MPa) had a significantly lower shear bond strength than either the EXL #547 self-etch primer (mean strength, 9.7 ± 3.7 MPa) or the phosphoric acid etch and the primer in the conventional adhesive system (mean strength, 10.4 ± 2.8 MPa).

DISCUSSION

The direct bonding of orthodontic brackets has revolutionized and improved the clinical practice of orthodontics. However, there is a need to improve the bonding procedure by saving time and also a need to minimize enamel loss without jeopardizing the ability to maintain a clinically useful bond strength. Although recent bonding systems are reliable in conservative dentistry, improvements in adhesive resins are still necessary to minimize technique sensitivity as well as to produce more durable materials. New materials should require a reduced number of steps and chair time. Traditionally, the use of acid etchants followed by a primer was an essential part of the bonding procedure of composite adhesives to allow good wetting and penetration of the sealant into the enamel surface.[5,6] The use of the new self-etching primers for orthodontic purposes has not been fully evaluated. In general, these new primers are thought to simplify the clinical handling of adhesive systems by combining the etchant and the primer in one application.[1,3,8–10] The earlier generation of acidic primers were selectively compatible with different adhesives and, as a result, either produced significantly lower bond strength or needed significantly more working time.[8]

The present study evaluated the use of three new self-etch primers (two already present on the market and the one still experimental) and compared them with a conventional adhesive bonding procedure. The findings indicated that one self-etch primer (EXL #547) provided a shear bond strength (mean strength, 9.7 ± 3.7 MPa) similar to that of the control group (mean strength, 10.4 ± 2.8 MPa) when used to bond orthodontic brackets to the enamel surface. However, the other two self-etch primers, Prompt L-Pop and One-Up Bond F, provided significantly lower shear bond forces (mean strength, 7.1 ± 4.4 MPa and 5.1 ± 2.5

MPa, respectively). These values are still considered to be clinically acceptable.[12] The average fluoride released by One-Up Bond F is 130 ppm/d during the first month, 50 ppm/d in the next two months, 20 ppm/d between six and 12 months, and 10 ppm/d between one and two years.[10] According to the manufacturer, these figures are somewhere between those obtained with cured-restorative glass ionomer cements and compomer restorative materials.[10]

It must be remembered that this is an in vitro study, and care should be taken in interpreting the results with respect to those that might be obtained in the oral environment. In addition, more research is needed to determine the shear bond strength of these new self-etch primers in the first half hour after bonding to simulate the time during which the initial archwires are tied.

CONCLUSION

By reducing the number of steps during bonding, clinicians are able to save time as well as reduce the potential for error and contamination during the bonding procedure. The present results indicated that the newly introduced self-etch primers containing both the enamel etchant and primer have the potential to be successfully used in bonding orthodontic brackets.

ACKNOWLEDGMENTS

The authors would like to express their gratitude to Morita USA Inc, 3M Unitek, and 3M ESPE for supplying the brackets and the adhesives for this study.

REFERENCES

1. Chigira H, Koike T, Hasegawa T, Itoh K, Wakumoto S, Hyakawa T. Effect of the self etching dentin primers on the bonding efficacy of dentine adhesive. *Dent Mater J.* 1989;8:86–92.
2. Nishida K, Yamauchi J, Wada T, Hosoda H. Development of a new bonding system. *J Dent Res.* 1993;72:137. Abstract 267.
3. Nakabayashi N. Dentinal bonding mechanisms. *Quint Int.* 1991; 22:73–74.
4. Gordan VV. *Acidic Primers in Dentin and Enamel—Shear Bond Strength and Microleakage* [master's thesis]. Iowa City, Iowa: University of Iowa; 1997.
5. Barkmeier WW, Erickson RL. Shear bond strength of composite to enamel and dentin using Scotchbond multi-purpose. *Am J Dent.* 1994;7:175–179.
6. Triolo PT Jr, Swift EJ Jr, Mudgil A, Levine A. Effects of etching time on enamel bond strengths. *Am J Dent.* 1993;6:302–304.
7. Britton JC, McInnes P, Weinberg R, Ledoux WR, Retief DH. Shear bond strength of ceramic orthodontic brackets to enamel. *Am J Orthod Dentofacial Orthop.* 1990;98:348–353.
8. Bishara SE, Gordan VV, VonWald L, Olson ME. Effect of an acidic primer on shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 1998;114:243–247.
9. Bishara SE, VonWald L, Laffoon JF, Warren JJ. Effect of using a self-etch primer/adhesive on the shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 2001;119:621–624.
10. Data from Tokuyama Tsukuba Research Laboratories. Tokyo, Japan.
11. Yamada T. Basic properties and clinical applications of the One-Up Bond F. *Dent Tribune.* 2000;7:1.
12. Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod.* 1979;2:171–178.