Shear Bond Strength of Orthodontic Brackets with Newly Developed Antibacterial Self-Etch Adhesive

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Abstract: Because the enamel adjacent to brackets may be affected by microorganisms, an antibacterial adhesive may be a useful choice to prevent and reduce demineralization. The purpose of this in vitro study was to determine the (1) shear bond strength of a self-etch and an antibacterial self-etch adhesive for orthodontic metal brackets and (2) bond failure interface of a self-etch and an antibacterial self-etch adhesive using a modified adhesive remnant index (ARI). Twenty-four defect-free premolars were randomly assigned into two groups. The teeth received the following treatments—group 1: Transbond Plus Self-Etching Primer + Transbond XT; group 2: antibacterial dentin bonding system (ABF) + Transbond XT. All samples were stored in deionized water at 37°C for 48 hours. Shear debonding tests were performed at a crosshead speed of five mm/min. The results in megapascals were (median, minimum, maximum) group 1: 8.53, 4.59, 12.63; group 2: 9.79, 4.01, 22.10, respectively. Mann-Whitney test revealed that the difference between the groups was not statistically significant ($P = .2$, $P > .05$). Failed brackets were examined by an optical microscope at 16× magnification to determine the bond failure interface using a modified ARI. The predominant mode of failure for both groups was at the bracket-adhesive interface. ABF may have sufficient mechanical properties and also an antibacterial effect that makes it a good choice for orthodontic bonding. (*Angle Orthod* 2005;75:843–848.)

Key Words: Bond strength; Antibacterial self-etch adhesive; Orthodontic bracket; ARI score

INTRODUCTION

The introduction of the acid-etch bonding technique by Buonocore in 1955 has led to dramatic changes in the practice of orthodontics.[1] By the late 1970s, the bonding of orthodontic brackets became an accepted clinical technique.[2,3] Bonded orthodontic brackets have advantages over bands in that they have no interproximal contacts, are both easier to place and to remove, and are more esthetic, hygienic, and less irritating to the gingiva.[4]

Orthodontic treatment with multibonded appliances imposes a significant caries risk.[5–8] Demineralization (decalcification) is a common side effect of fixed appliance orthodontic treatment.[9] It occurs when the pH of the oral environment favors diffusion of calcium and phosphate ions out of enamel and is reported to occur in anywhere from 2% to 96% of orthodontic patients.[10] The components of the appliance and the bonding materials often promote plaque accumulation with subsequent acid production leading to decalcification and an alteration in the appearance of the enamel surface.[5,6,11]

The development of fluoride-releasing composites and glass ionomer cements for bracket bonding has attracted considerable interest because they may inhibit the decalcification of the enamel around the brackets by offering fluoride delivery to the environment.[12–17] Remineralization by release of fluoride is important, but the antibacterial property of fluoride is a direct strategy to eliminate the cause of dental caries.[18]

Under these circumstances, bioactive adhesive systems that possess antibacterial effects or intensive remineralization ability are considered beneficial and capable of producing superior clinical performances.[19] Recently, an experimental fluoride-releasing antibac-

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TABLE 1. Materials and Application Procedures[a]

Materials	Components	Chemical Composition	Steps of Application
Experimental ABF (Kuraray)	Primer	MDP[a], MDPB[b], HEMA[c], water, hydrophilic dimethacrylate	Apply and leave for 20 s, air dry
	Bond	MDP, Bis-GMA[d], HEMA, microfiller, surface-treated sodium fluoride	Apply bond and light cure for 10 s
Transbond XT Self-Etching Primer	Primer & Bond	Fluoride, no filler, methacrylate ester derivative	Rub enamel for 3 s, air dry gently
Transbond XT Light Cure Adhesive	Paste	Quartz silica, Bis-GMA, bisphenol A bis (2-hydroxyethyl ether) dimetacrylate	Light cure for 20 s

[a] MDP indicates 10-methacryloyloxydecyldihydroges phosphate.
[a] MDPB indicates methacryloxydodecyl-pyridinium bromide.
[c] Hema indicates 2-hydroxyethylmethacrylate.
[d] Bis-GMA indicates disphenol A diglycidyl ether dimethacrylate.

terial bonding agent was developed by combining the physical advantages of dental adhesive technology and an antibacterial effect.

The purposes of this in vitro study were (1) to determine the shear bond strength of the brackets bonded with a fluoride-releasing, self-etching adhesive system and an experimental antibacterial, self-etching adhesive system and (2) to determine the mode of failure of the brackets as reported by a modified adhesive remnant index (ARI).

MATERIALS AND METHODS

Twenty-four human premolars with intact buccal enamel and free of caries and cracks were extracted for orthodontic purposes and stored in distilled water. The teeth were cleaned and polished with pumice and rubber prophylactic cups for 10 seconds, embedded in methylmethacrylate, and randomly divided into two groups. The teeth received the following treatments:

• Group 1: A fluoride-releasing, self-etching adhesive system (n = 12): Transbond Plus Self-Etching Primer (3M Unitek) + Transbond XT (3M Unitek, Monrovia, California)
• Group 2: The experimental, fluoride-releasing, antibacterial, self-etching adhesive system (n = 12): Experimental antibacterial dentin bonding system (ABF) (Kuraray, Osaka, Japan) + Transbond XT (3M Unitek)

Orthodontic metal brackets (Ormco Series 2000; first and second premolars w/hook) were used in this study. The average bracket base surface area was 9.63 mm[2]. The dimensions of the bracket were measured by a caliper compass and scanned. The scanned image was turned into a vectorial construction by Autocad software program. The measured dimensions were double-checked on the PC, and a three-dimensional, solid model was achieved by the Pro-Engineer program. Finally, the surface area of the bracket was calculated by this software. The brackets

were bonded to the teeth according to the manufacturers' directions (Table 1).

All samples were stored in deionized water at 37°C for 48 hours. A universal test machine (Zwick Test Machine, Zwick GmbH & Co, Ulm, Germany) was used for the shear bond test at a crosshead speed of five mm/min. Each tooth was oriented so that its facial surface was parallel to the direction of force during the shear strength testing. Force was directly applied to the bracket-tooth interface by a one end flattened steel rod. The load at bracket failure was recorded by a PC connected to the Zwick test machine. The shear bond strength values were calculated in megapascals by dividing the force by the area of the bracket base. One sample from each group was lost during the debonding test. The lost samples were excluded from all statistical analyses.

After debonding, the teeth and brackets were examined under 16× magnification by an optical microscope (Leica MS5, Singapore, Singapore). Any adhesive that remained on the bracket after bracket removal was assessed according to a modified ARI and scored with respect to the amount of resin material that remained on the bracket surface.[20] The ARI scale has a range of 5 to 0 (5 = 100% of adhesive left on bracket, 4 = 100–75% adhesive left on bracket, 3 = 75–50% of adhesive resin left on the bracket, 2 = 50–25% of adhesive left on the bracket, 1 = less than 25% of adhesive left on the bracket, 0 = no adhesive left on the bracket). One sample from each group was lost during the ARI scoring.

Median, minimum, and maximum values were calculated for each of the two test groups. The Mann-Whitney test was used for the statistical analysis. Significance was determined at a probability value of $P <$ .05.

RESULTS

The median, minimum, and maximum values for shear bond strength of the two groups are presented

TABLE 2. Shear Bond Strength of the Adhesives

	n	Median (MPa)	Minimum (MPa)	Maximum (MPa)
Group 1	11	8.53	4.59	12.63
Group 2	11	9.79	4.01	22.10

in Table 2. There was no statistical significance between two groups ($P = .2$; $P > .05$). The brackets bonded with either of the two adhesives can withstand equal amounts of force during the shear bond test.

The frequency distribution of the ARI score for groups is shown in Table 3. The predominant mode of bracket failure for both groups was at the bracket-adhesive interface leaving less than 25% of the adhesive on the bracket base.

DISCUSSION

The universal testing machine is a stable and rigid device capable of producing pure shear debonding forces, whereas a clinician introduces a combination of shear, tensile, and torsional forces when performing in vivo debonding. In addition, the rate of loading for a universal testing machine is constant, whereas the rate of loading for in vivo debonding is not standardized or constant. Thus, the minimum in vitro bond strength required for clinical reliable orthodontic debonding procedures still varies, depending on factors such as the adhesive systems used, bracket base design, enamel morphology, appliance force systems, and the clinician's technique.[21] However, it is obvious that the in vitro studies provide a guide for the clinician in the selection of the bracket/adhesive choice to be used in vivo.[22]

Phosphoric acid etching produces a roughened enamel surface by dissolving calcium components and forms enamel resin tags. Although the enamel etching technique is a useful and accepted orthodontic procedure for bonding orthodontic brackets, there is a need to improve the ability to maintain clinically useful bond strengths while minimizing the enamel loss.[23] Self-etching primers function both as etching agents and primers.

Rinsing of the enamel after application of the self-etching primer is not required. Thus, the use of a self-etching primer reduces the number of clinical steps and saves clinical operation time because separate acid etching and water rinsing steps are eliminated and application requires simply drying with air. The self-etching adhesive systems produce high bond strengths to human coronal dentin and ground enamel surfaces. These materials seem to be very promising for further clinical applications, and the results are very encouraging for the clinical success of these simplified adhesive systems.[24] Moreover, phosphoric acid etching has been blamed for decalcification and the development of white-spot lesions around bonded orthodontic appliances.[25,26]

The experimental self-etching adhesive system ABF has 12-methacryloyloxydodecyl-pyridinium bromide (MDPB), which is an antibacterial monomer incorporated in antibacterial adhesives. It was developed by combining the antibacterial agent quaternary ammonium and a methacryloyl group.[27–29] Imazato et al[30,31] have studied the use of MDPB since 1995 and reported incorporation of MDPB into the self-etching primer and adhesive resin. MDPB copolymerizes with other monomers after curing, and the antibacterial agent is covalently bonded to the polymer network. The immobilized agent does not leach out from the material but acts as a contact inhibitor against the bacteria that attach to the surface.[18] They have reported findings concerning in vitro antibacterial activity, bonding ability, cytotoxicity and pulpal response of MDPB-containing self-etching primer/adhesive.[30–36] In addition, they confirmed that an MDPB-containing primer could show antibacterial effects in vivo using animal models, and validated the usefulness of the antibacterial adhesive systems.[37]

Unlike its fluoride-release effect, the antibacterial activity of MDPB may not extend around the bracket thus, producing a potential limitation. The immobilized agent may be effective when bacteria contact the surface, however, studies in restorative dentistry have demonstrated that polymerization shrinkage and marginal leakage occurs between the tooth surface and the resin composite after polymerization. In addition, polymerization shrinkage increases when the filler content of the composite decreases. Although there is not much study on the microleakage of orthodontic brackets in the literature, clinicians usually face the problem of white-spot lesions around and beneath the orthodontic brackets.[5]

The bond strength of adhesives should be sufficient

TABLE 3. The Frequency Distribution of ARI Scores Among Groups[a]

	n	0	1	2	3	4	5
Group 1	10	4	5	0	1	0	0
Group 2	10	3	3	2	1	0	1

[a] ARI indicates adhesive remnant index.

to withstand the forces of mastication, the stress exerted by the archwires, and patient abuse. At the same time, the bond strength should be at a level to allow for bracket debonding without causing damage to the enamel surface. Various studies have suggested bond strengths ranging from 2.8 to 10 MPa as being adequate for clinical situations.[38–41] According to Reynolds, adequate bond forces for routine orthodontic treatment range between 5.9 to 7.8 MPa.[40] The maximum bond strength should be less than the breaking strength of enamel, which is about 14 MPa.[42,43]

The results of the debonding test showed that the median shear bond strength of the two adhesives lay between 2.8 and 10 MPa. However, there is a large difference between the minimum and maximum values in the second group, whereas the maximum value is 22.10 MPa. This value exceeds the range of shear strength of enamel and may thus cause crazing in the enamel surface during the debonding procedure.

The manufacturers usually recommend etching unground enamel before using self-etching adhesives to increase the bond strength because the use of primer alone may not sufficiently condition the uncut enamel. However, the results of this study may show that etching before priming is not necessary because the resultant bond strength is quite enough for orthodontic purposes, and decalcification by phosphoric acid may be avoided.

The objectives of debonding are to remove the attachment and all the adhesive resin from the tooth and to restore the surface as closely as possible to its pretreatment condition. There are several factors involved in this procedure, the most important of which are the instruments used for bracket removal, the armamentarium for resin removal, and the type of adhesive used.[4]

Many studies have shown that when self-etching primers are used, the degree of penetration by the adhesive to the etched enamel is less than with the use of a conventional acid-etch technique. The more deeply the enamel surface is penetrated by the adhesive, the greater the penetration of the adhesive and the greater the risk of damage to the enamel during debonding.[44]

The sites of failure within the bracket-adhesive-enamel complex can occur within the bracket, between the bracket and the adhesive, within the adhesive, and between the tooth surface and the adhesive.[45] A modified ARI has been developed to quantify the amount of adhesive that remains on the bracket after a bracket base debond.[20]

Martin and Garcia-Godoy[46] have stated that high shear strength in orthodontics is not necessarily a beneficial property of an adhesive because enamel can be lost during the debonding procedures as well as during the cleanup process of residual resin removal.

There are two basic opinions on the remnant adhesive on the teeth surface after bracket debonding. One opinion mainly favors the bracket-adhesive interface failure leaving the adhesive resin mainly on the enamel surface.[4,47] This is important when a heavy filled resin is used to bond orthodontic attachments to the enamel because the micro porosities created by etching are filled with the resin and provide mechanical retention. This is consistent with the finding of Bennett et al.[48] In our study, the dominant ARI score is 1, which means 25% or less adhesive is left on the bracket. Our findings mainly support this opinion.

The second opinion favors the failure at the enamel-adhesive resin interface because there will be less adhesive left to remove from the enamel surface after debonding.[49] This is contradictory to our findings. From our point of view enamel fracture and crazing may be seen during bracket debonding especially with ceramic brackets.[50] This is of clinical significance because the concentration of fluoride is greatest at the surface of the enamel.[51] However, Martin and Garcia-Godoy[46] suggested that a weaker adhesive with a lower strength value might be preferable to increase failure or bracket debonding at the resin enamel interface so that minimal clean up effort will be needed and no damage to the enamel will occur.

CONCLUSIONS

- Experimental antibacterial dentin bonding system (ABF) may be a prospective candidate for bonding brackets because it seems to have sufficient mechanical properties.
- However, further research is needed to clarify whether the materials are sufficiently effective in inhibiting bacterial activity under in vivo conditions.

REFERENCES

1. Zarrinnia K, Eid NM, Kehoe MJ. The effect of different debonding techniques on the enamel surface: an *in vitro* qualitative study. *Am J Orthod Dentofacial Orthop.* 1995;108: 284–293.
2. Thanos CE, Munholland T, Caputo AA. Adhesion of mesh base direct bonding brackets. *Am J Orthod.* 1979;75:421–430.
3. Gorelick L. Bonding metal brackets with a self-polymerizing sealant-composite: a 12-month assessment. *Am J Orthod.* 1977;71:542–553.
4. Proffit W. *Contemporary Orthodontics.* St Louis, Mo: CV Mosby; 1986:287.
5. Årtun J, Brobakken BO. Prevalence of carious white spots after orthodontic treatment with multibonded appliances. *Eur J Orthod.* 1986;8:229–234.
6. O'Reilly MM, Featherstone JD. Demineraliztion and remineralization around orthodontic appliances: an *in vitro* study. *Am J Orthod Dentofacial Orthop.* 1987;92:33–40.

7. Zachrisson BU, Zachrisson S. Caries incidence and orthodontic treatment with fixed appliances. *Scand J Dent Res.* 1971;79:183–192.

8. Øgaard B. Prevalence of white spot lesions in 19-year-olds: a study on untreated and orthodontically treated persons 5 years after treatment. *Am J Orthod Dentofacial Orthop.* 1989;96:423–427.

9. Millett DT, Nunn JH, Welbury RR, Gordon PH. Decalcification in relation to brackets bonded with glass ionomer cement or a resin adhesive. *Angle Orthod.* 1999;69:65–70.

10. Mitchell L. Decalcification during orthodontic treatment with fixed appliances—an overview. *Br J Orthod.* 1992;19:199–205.

11. Zachrisson BU, Brobakken BO. Clinical comparison of direct versus indirect bonding with different bracket adhesives. *Am J Orthod.* 1978;74:62–78.

12. Sonis AL, Snell W. An evaluation of a fluoride-releasing visible light-activated bonding system for orthodontic bracket placement. *Am J Orthod Dentofacial Orthop.* 1989;95:306–311.

13. Underwood ML, Rawis HR, Zimmermann BF. Clinical evaluation of a fluoride-exchanging resin as an orthodontic adhesive. *Am J Orthod Dentofacial Orthop.* 1989;94:96–99.

14. Chan DCN, Swift EJ, Bishara SE. *In vitro* evaluation of a fluoride-releasing orthodontic resin. *J Dent Res.* 1990;69:1576–1579.

15. Øgaard B, Rezk-Lega F, Ruben J, Arends J. Cariostatic effect and fluoride release from a visible light-curing adhesive for bonding of orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 1992;101:303–307.

16. Mitchell L. An investigation into the effects of fluoride-releasing adhesive on the prevalence of enamel surface changes associated with directly bonded orthodontic attachments. *Br J Orthod.* 1992;19:207–214.

17. Turner PJ. Clinical evaluation of a fluoride-containing orthodontic material. *Br J Orthod.* 1992;201:307–313.

18. Imazato S. Antibacterial properties of resin composites and dentin bonding systems. *Dent Mater.* 2003;19:449–457.

19. Imazato S. Progress of adhesive systems as a cornerstone of minimal intervention dentistry. In: Yoshiyama M, Nishitani Y, Nakabo S, Cox CF, eds. *Modern Trends in Adhesive Dentistry Proceedings of the Adhesive Dentistry Forum of 2001 in Okuyama, Japan.* Tokyo, Japan: Kuraray Medical Inc: 2002:1–13.

20. Shammaa I, Ngan P, Kim H, Kao E, Gladwin M, Gunel E, Brown C. Comparison of bracket debonding force between two conventional resin adhesives and resin-reinforced glass ionomer cement: an *in vitro* and *in vivo* study. *Angle Orthod.* 1999;69:463–469.

21. Greenlaw R, Way DG, Khadry AG. An *in vitro* evaluation of a light-cured resin bonding systems. *Am J Orthod Dentofacial Orthop.* 1989;96:214–220.

22. Pickett KL, Sadowsky PL, Jacobson A, Lacefield W. Orthodontic *in vivo* bond strength: comparison with *in vitro* results. *Angle Orthod.* 2001;71:141–148.

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

24. Kiremitci A, Yalcin F, Gokalp S. Bonding to enamel and dentin using self-etching adhesive systems. *Quintessence Int.* 2004;35:367–370.

25. Gorelick L, Geiger AM, Gwinnett AJ. Incidence of white spot formation after bonding and banding. *Am J Orthod.* 1982;81:93–98.

26. Øgaard B, Rølla G, Arends J. Orthodontic appliances and enamel demineralization. Part 1: lesion development. *Am J Orthod Dentofacial Orthop.* 1988;94:68–73.

27. Imazato S, Torii M, Tsuchitani Y, McCabe JF, Russell RRB. Incorporation of bacterial inhibitor into resin composite. *J Dent Res.* 1994;73:1437–1443.

28. Imazato S, McCabe JF. Influence of incorporation of antibacterial monomer on curing behavior of a dental resin composite. *J Dent Res.* 1994;73:1641–1645.

29. Imazato S, Tarumi H, Kato S, Ebisu S. Water sorption and color stability of composites containing the antibacterial monomer MDPB. *J Dent Res.* 1999;27:279–283.

30. Imazato S, Kinomoto Y, Tarumi H, Torii M, Russel RRB, McCabe JF. Incorporation of antibacterial monomer MDPB in dentin primer. *J Dent Res.* 1997;76:768–772.

31. Imazato S, Kinomoto Y, Tarumi H, Ebisu S, Tay FR. Antibacterial activity and bonding characteristics of an adhesive resin containing antibacterial monomer MDPB. *Dent Mater.* 2003;19:313–319.

32. Imazato S, Imai T, Russell RRB, Torii M, Ebisu S. Antibacterial activity of cured dental resin incorporating the antibacterial monomer MDPB and an adhesion-promoting monomer. *J Biomed Mater Res.* 1998;39:511–515.

33. Imazato S, Ehara A, Torii M, Ebisu S. Antibacterial activity of dentin priming containing MDPB after curing. *J Dent.* 1998;26:267–271.

34. Tarim B, Hafez AA, Akimoto N, Imazato S, Cox CF. Pulp response of direct capped exposures at 6 & 7 months in monkeys [Special Issue]. *J Dent Res.* 1998;77:800.

35. Imazato S, Tarumi H, Ebi N, Ebisu S. Cytotoxic effects of composite restorations employing self-etching primers or experimental antibacterial primers. *J Dent.* 2000;28:61–76.

36. Imazato S, Torii Y, Takatsuka T, Inoue K, Ebi N, Ebisu S. Bactericidal effect of dentin primer containing antibacterial monomer methacryloyloxydodecyl-pyridinium bromide (MDPB) against bacteria in human carious dentin. *J Oral Rehabil.* 2001;28:314–319.

37. Kaneko T, Imazato S, Ebi N, Kuramoto A, Noiri Y, Ebisu S. *In vivo* antibacterial effect of dentin primer incorporating MDPB [Special Issue]. *J Dent Res.* 2001;80:659.

38. Keizer S, Ten Cate JM, Arends J. Direct bonding of orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 1976;69:318–327.

39. Lopez JI. Retentive shear strengths of various bonding attachment bases. *Am J Orthod Dentofacial Orthop.* 1980;77:669–678.

40. Reynolds I. A review of direct orthodontic bonding. *Br J Orthod.* 1975;2:171–178.

41. Buonocore MG. Principles of adhesive retention and adhesive restorative materials. *J Am Dent Assoc.* 1963;67:382–391.

42. Retief DH. Failure at the dental adhesive-etched enamel interface. *J Oral Rehabil.* 1974;1:265–284.

43. Bowen RL, Rodriguez MS. Tensile strength and modulus of elasticity of tooth structure and several restorative materials. *J Am Dent Assoc.* 1962;64:378–387.

44. Bishara SE, Von Wald L, Laffoon JF, Warren J. Effect of a self-etch primer/adhesive on the shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 2001;119:621–624.

45. Littlewood SJ, Mitchell L, Greenwood DC, Bubb NL, Wood DJ. Investigation of a hydrophilic primer for orthodontic bonding: an *in vitro* study. *J Orthod.* 2000;27:181–186.

46. Martin S, Garcia-Godoy F. Shear bond strength of orthodontic brackets cemented with a zinc oxide-polyvinyl cement. *Am J Orthod Dentofacial Orthop.* 1994;106:615–620.

47. Bishara SE, Ajlouni R, Laffoon JF. Comparison of the shear bond strength of 2 self-etch primer/adhesive systems. *Am J Orthod Dentofacial Orthop.* 2004;125:348–350.

48. Bennett CG, Shen C, Waldron JH. The effects of debonding on the enamel surface. *J Clin Orthod.* 1984;18:330–334.

49. Bishara SE, Von Wald L, Laffoon JF. Effect of altering the type of enamel conditioner on the shear bond strength of a resin-reinforced glass ionomer adhesive. *Am J Orthod Dentofacial Orthop.* 2000;118:288–294.

50. Britton C, 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.

51. Retief DH, Sadowsky PL. Clinical experience with the acid-etch technique in orthodontics. *Am J Orthod Dentofacial Orthop.* 1975;68:645–654.