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Published version. *The Angle Orthodontist,* Vol. 78, No. 2 (March 2008): 339-344. DOI.

Original Article

Bond Strength of an Amorphous Calcium Phosphate–Containing Orthodontic Adhesive

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ABSTRACT

Objective: To determine whether an amorphous calcium phosphate (ACP)-containing adhesive has an acceptable level of shear bond strength to be used as an orthodontic adhesive.

Materials and Methods: Sixty extracted premolars were randomly divided into three groups for orthodontic bonding. Group 1 used a composite resin adhesive (Transbond XT), group 2 was bonded with an ACP-containing adhesive (Aegis Ortho), and group 3 used a resin-modified glass ionomer (Fuji Ortho LC). All bonded teeth were stored in distilled water at 37°C for 40 ± 2 hours prior to debonding. Shear bond strength and adhesive remnant index (ARI) were recorded for each specimen.

Results: The mean shear bond strengths for the three test groups were: group 1 (15.2 ± 3.6 MPa), group 2 (6.6 ± 1.5 MPa), and group 3 (8.3 ± 2.8 MPa). A one-way analysis of variance showed a significant difference in bond strengths between the groups. A post hoc Tukey test showed group 1 to be significantly ($P < .001$) greater than groups 2 and 3. A Kruskal-Wallis test and a Mann-Whitney *U*-test showed groups 1 and 3 exhibited lower ARI scores than group 2, but a majority of specimens in each group had greater than 50% of the cement removed along with the bracket during debonding.

Conclusions: The ACP-containing adhesive demonstrated a low, but satisfactory bond strength needed to function as an orthodontic adhesive.

KEY WORDS: Bond strength; Amorphous calcium phosphate; Adhesive

INTRODUCTION

White spot lesions displayed on the enamel surface are a result of organic acids, produced by cariogenic bacteria housed in retained areas of dental plaque. Full orthodontic treatment frequently spans approximately a 2-year period, and at this time 50% of patients display some form of clinical white spots.[1] Lesions of significant depth (75 μm) can develop in as little as 4 weeks, a shorter time period than many orthodontic appointment intervals of 6 to 10 weeks.[2,3] Decalcified lesions are not only unesthetic, but they may become irreversible and lead to cavitated lesions. For these reasons, white spot lesions are of great concern to orthodontics, and advancements in orthodontic adhesive materials serve as one possible avenue to prevent this occurrence.

Amorphous calcium phosphate (ACP) has the properties of both a preventive and restorative material that justify its use in dental cements, sealants, composites, and more recently, orthodontic adhesives. ACP-filled composite resins have been shown to recover 71% of the lost mineral content of decalcified teeth.[4] One ACP-containing adhesive, Aegis Ortho (The Bosworth Co, Skokie, Ill), has been marketed for use as a light-cured orthodontic adhesive with similar properties to previously used resins. The premise behind this product is that during a carious attack around orthodontic brackets, at or below pH 5.8, hydroxyapatite (HAP) is leached from the enamel surface. At this low pH, ACP is capable of being broken down and releasing supersaturating levels of Ca^{+2} and PO_4 ions.[5] These concentrations are conducive to the formation of HAP, which in turn can be used by the tooth for remineral-

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Accepted: April 2007. Submitted: February 2007.

ization.[6] This elevated state can be maintained for a considerable time, offering a promising antagonist to demineralization, and can promote the prevention of future white spots throughout orthodontic treatment.

ACP-containing orthodontic adhesives are claimed to reduce the prevalence of white spots around orthodontic appliances. However, to even be considered for clinical use, they must first provide satisfactory shear bond strength. Therefore, the aim of this study was to determine whether Aegis Ortho adhesive provided an acceptable level of shear bond strength to function as an orthodontic adhesive. This product was compared with the commonly used orthodontic adhesives/cements, Transbond XT and Fuji Ortho LC. Transbond XT adhesive, a composite resin, was chosen as one of the controls due to its popularity and common use in other bond strength studies.[7,8] Similar to the claims made by the ACP-adhesive manufacturer, the use of resin-modified glass ionomer cement (Fuji Ortho LC) has been said to be more effective in preventing white spot lesions than conventional composite resins.[9] Using this cement as another comparative group allows comparison of adhesives marketed for the purpose of white spot prevention. The efficacy of the ACP-containing adhesive in preventing demineralization was not the focus of this study, but if proven to have adequate bond strength, this feature will be explored in future research.

MATERIALS AND METHODS

Sixty extracted human premolars, absent of restorations and caries, were collected and stored in distilled water at room temperature. The teeth remained in distilled water at all times except when brackets were being bonded and debonded. The teeth were randomly divided into three groups, all containing 20 specimens. Group 1 was bonded using Transbond XT (3M/Unitek Corp, Monrovia, Calif), group 2 was bonded with Aegis Ortho (Bosworth), and group 3 used Fuji Ortho LC (GC America Inc, Alsip, Ill). The direct bonding protocol provided by the manufacturer was followed for each individual adhesive.

Prior to bonding, the teeth were sectioned 2 to 3 mm below the cementoenamel junction using a separating disk and a lab hand piece to help minimize heat. Before bonding, all teeth were cleaned using coarse, oil-free pumice with a rubber prophylaxis cup, rinsed with water, and dried for 10 seconds using an air-water syringe. Etchant (Transbond XT etching gel, containing 35% phosphoric acid from 3M/Unitek) was then applied to the cleaned area of the tooth for 15 seconds, rinsed for 10 seconds, and dried with an air-water syringe for 20 seconds. This etchant was used for all three groups to provide consistency in tooth preparation. In the instance of group 3, the tooth was left moist to remain in compliance with the recommended bonding protocol of the manufacturer. Universal bicuspid brackets, Victory Series (3M/Unitek), with a 0.022-inch slot and 0° of tip and torque were used in this experiment. This bracket is a stainless steel miniature mesh twin bracket containing a 10 mm^2 base surface area. All bonding was performed by a single operator. Published guidelines for bond strength testing were followed when possible to allow for past and future data comparisons.[10]

Bonding

For group 1, each bracket was bonded one at a time using a direct bond technique. A thin layer of Transbond XT light-cured primer was applied to the tooth and light cured for 10 seconds. Transbond XT adhesive was applied to the bracket base, and the bracket was placed onto the tooth in the center of the crown, with the center of the bracket over the long axis of the tooth. The excess adhesive was removed with a hand instrument, and the bracket was cured for 10 seconds from the distal and 10 seconds from the mesial. The same instructions as in group 1 were followed for the adhesives used in group 2 (Aegis Ortho) and group 3 (Fuji Ortho LC), except neither required the use of a primer.

Mounting and Shear Bond Testing Procedure

After the brackets were bonded, the teeth were mounted in acrylic and stored in fresh distilled water at 37°C for 40 hours (±2 hours) prior to debonding. A storage time of 40 hours was chosen since short times in excess of 24 hours are not critical[10] and also to remain consistent with previous studies using Transbond XT as a control.[8,11] An Instron universal testing machine (Instron Corp, Canton, Mass) was used to debond the brackets using a shear load applied to the bracket at a crosshead speed of 0.1 mm/min. Specimens were positioned in the Instron machine in a manner such that the loading force was directed parallel to the long axis of the tooth and contact was made as close to the bracket/tooth interface as possible. For each specimen, the maximum load to debond was recorded.

Adhesive Remnant Index Classification

Following debonding teeth and brackets were analyzed under a Spencer optical stereomicroscope using external illumination and given a score according to the adhesive remnant index (ARI). This test has four possible outcomes:

0, no adhesive left on the tooth

Table 1. Shear Bond Strength

Group	Bond Strength, MPa				
	Mean	Standard Deviation	Minimum	Maximum	Range
1-Transbond XT	15.2*	3.6	8.5	22.2	13.7
2-Aegis Ortho	6.6**	1.5	4.4	10.1	5.7
3-Fuji Ortho LC	8.3**	2.8	2.5	13.9	11.4

* Group 1 was significantly different ($P < .001$) from groups 2 and 3.
** Groups 2 and 3 were not significantly different ($P > .05$) from each other.

1, less than half of the adhesive left on the tooth
2, more than half of the adhesive left on the tooth
3, all of the adhesive left on the tooth with a distinct impression of the bracket mesh

Statistical Analysis

Differences in shear bond strength between the three groups were analyzed using a one-way analysis of variance (ANOVA) and post hoc Tukey test at a $P \leq .05$ level of confidence. A Weibull analysis was performed to determine bond strength reliability at specific load values. In addition, a Kruskal-Wallis test followed by a Mann-Whitney *U*-test was used to determine any statistically significant differences in the ARI scores between the groups. The statistical analysis was performed using SPSS 12.0.1 (SPSS Inc, Chicago, Ill).

RESULTS

The mean, standard deviation, minimum/maximum, and range of shear bond strengths for the three groups are displayed in Table 1. ANOVA and the post hoc Tukey test revealed that group 1 was significantly different from groups 2 and 3 ($P < .001$). Groups 2 and 3 were not significantly different from each other ($P > .05$).

In evaluating the usefulness of a bonding material, the lower values in a distribution of bond strengths are frequently more important clinically compared to mean bond strength since these lower values are more likely to result in clinical debonds.[11] The Weibull analysis is a survival analysis tool that has been used in orthodontic research to evaluate the distribution of bond strengths. The Weibull curves for the three groups are displayed in Figure 1. Additionally, the characteristic bond strength, Weibull modulus, and shear bond strengths at 10% and 90% probability of failure are displayed in Table 2.

The ARI scores are displayed in Table 3. A statistical difference was found with a Kruskal-Wallis test ($P < .001$). Mann-Whitney *U*-tests showed groups 1 and 3 were not significantly different ($P = .057$). Group 2 displayed a significantly greater ARI score compared to groups 1 and 2 ($P < .01$).

DISCUSSION

Tavas and Watts reported that shear/peel strengths of direct bonded adhesives should develop to 4 kg in 5 minutes and 6 kg in 24 hours.[12] For the brackets used in this study, these force values correspond to 3.9 and 5.9 MPa, respectively. Reynolds stated that successful clinical bonding has been recorded with adhesives giving in vitro tensile bond strengths of 4.9 MPa.[13] The shear bond strengths for groups 1, 2, and 3 were 15.2 ± 3.6, 6.6 ± 1.5, and 8.3 ± 2.8 MPa, respectively. All three groups possessed mean shear bond strengths above the amount recommended by Tavas and Watts to perform as an orthodontic bracket adhesive. The ACP-containing adhesive presented with the lowest mean shear bond strength; however, the statistical analysis showed that there was no significant difference between this adhesive and the resin-modified glass ionomer cement which has been used in orthodontic practice for several years.

Results for groups 1 and 3 concur with many other studies investigating the bond strength of these adhesives. Transbond XT displayed a similar mean bond strength and ARI score when compared with previous publications that used the same bracket type.[7,11] Additionally, as shown in the present study, Transbond XT has been found to have a superior bond strength to Fuji Ortho LC in the majority of studies that have investigated them together.[14–17] Regardless of ranking, these same studies concluded that Fuji Ortho LC exhibited sufficient bond strength to be used as an orthodontic adhesive. Only a few articles have investigated the incorporation of calcium phosphates into orthodontic adhesives. Dunn[18] examined the bond strength of the same amorphous calcium phosphate adhesive used in this study and observed that brackets failed at a significantly lower force level when compared to a traditional composite resin adhesive, as was found in the current study. Kawabata et al[19] investigated the bond strength of a resin cement containing varying amounts of α-tricalcium phosphate and concluded the formulations possessed a clinically acceptable bond strength.

As mentioned previously, the mean shear bond strength may not be the most useful indicator of performance for evaluating orthodontic adhesives. Of greater significance to the clinician are the weaker values in the collection, because these represent instances which may result in the possibility of early clinical bond failure. This aspect is evaluated in the Weibull analysis in Figure 1 and Table 2. Group 2 showed the lowest characteristic strength and bond strength at 10% and 90% probability of failure. Consistent with the mean shear bond strength data, group 3 was not much

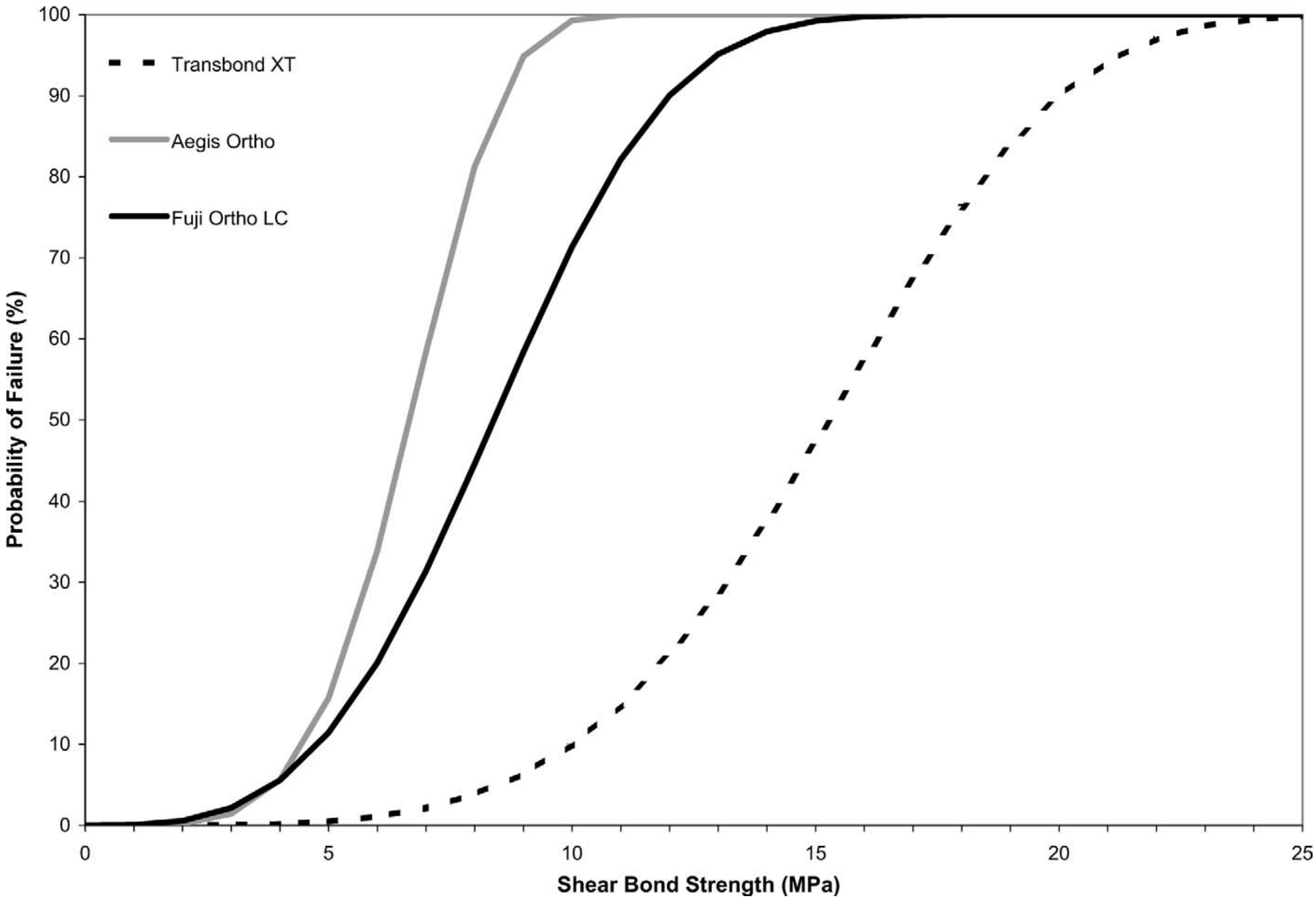

Figure 1. Weibull curves for the shear bond strength of the three groups.

greater in these parameters than group 2 and was far less than group 1.

Although derived from in vitro data, Figure 1 indicates groups 2 and 3 have a greater than 10-fold probability of failing at the critical 4.9–5.9 MPa bond strengths mentioned above. Similarly, Table 2 shows the bond strengths at a 10% probability of failure for groups 2 and 3 are lower than those recommended bond strength values. This suggests brackets bonded with the ACP-containing adhesive (or resin-modified glass ionomer) may have a greater chance of clinically debonding. Clinical studies with Fuji Ortho LC have been inconclusive in this regard. Gaworski et al[20] found a greater percentage of debonds with Fuji Ortho LC compared to a resin adhesive, while others found similar clinical debond rates between Fuji Ortho LC and several resin adhesives.[21–23] No clinical studies have examined the survival rate of brackets bonded with ACP-containing adhesives. It should be stressed, however, that if this adhesive is found to have remineralization capabilities, it may be acceptable to some clinicians and patients to have a slightly greater chance of debonding if it prevents white spots.

One aspect uncovered in the Weibull analysis and statistical analysis was the consistency of bond strength with the ACP-containing adhesive. This is evidenced by the lowest standard deviation (1.5 MPa) and greatest Weibull modulus (4.9) being for group 2 compared to those for group 1 (3.6 and 4.5, respectively) and group 3 (2.8 and 3.4, respectively). The

Table 2. Weibull Modulus and Characteristic Strength Results

Group	Weibull Modulus, β	Characteristic Strength, α	Shear Bond Strength at 10% Probability of Failure, MPa	Shear Bond Strength at 90% Probability of Failure, MPa
1-Transbond XT	4.5	16.6	10.0	20.0
2-Aegis Ortho	4.9	7.2	4.5	8.5
3-Fuji Ortho LC	3.4	9.4	4.8	12.0

Table 3. Adhesive Remnant Index (ARI) Scores by Group

Group	ARI Scores*			
	0	1	2	3
1-Transbond XT	11	5	3	1
2-Aegis Ortho	1	10	9	0
3-Fuji Ortho LC	16	4	0	0

* There was no significant difference ($P > .05$) between groups 1 and 3; however, group 2 was significantly different ($P < .01$) from groups 1 and 3.

greater the Weibull modulus, the more reliable the data or the closer the bond strengths are grouped together. It should be noted, however, that the bond strength range for the ACP-containing adhesive was lower than that of the other groups, perhaps due to its lesser maximum bond strength, and this may partially account for its low standard deviation and high Weibull modulus. Nonetheless, the ACP-containing adhesive produced a consistent bond, a desirable aspect to clinicians.

In orthodontics, less adhesive left on the tooth after debonding corresponds to less work and time spent by the orthodontist in removing it. A lower ARI score is favorable in this situation and all three groups presented with a majority of specimens in the 0–1 range, thus a majority of the adhesive was removed with the bracket during the process of debonding. Group 2 did have a significantly ($P < .05$) greater ARI score compared to the other two groups. Therefore, the clinician would have to spend a little more time removing the ACP-containing adhesive from the tooth. Additionally, this difference in ARI scores suggests the ACP-containing adhesive exhibited more of a cohesive failure compared to the composite resin and resin-modified glass ionomer adhesives.

Several different modalities have been employed to combat the problem of demineralization or white spot formation during orthodontic treatment. Derks et al[24] concluded that the most effective means of preventing white spots is through the use of a high fluoride (1500–5000 ppm) gel and toothpaste used daily during orthodontic treatment. Another study indicated daily brushing with a fluoridated dentifrice along with daily rinsing with fluoride will help prevent demineralization.[25] Both of these studies are relevant if compliance is not an issue. However, that is not always the case. A compliance rate of only 13% was achieved from patients when they were asked to use fluoride mouth rinse daily during orthodontic treatment.[26] Glass ionomer cement has been scrutinized in numerous studies and the results for and against its use have been presented.[27,28] This has prompted companies to develop new adhesives.

Being an in vitro bond strength study, caution is advised in extrapolating the results of the present study to the clinical situation. Clinically, the adhesives are subject to stresses, temperature fluctuations, variable electrolytes, microorganisms, and other factors that may affect performance. Kawabata et al[19] showed α-tricalcium phosphate incorporated resins, thermal cycled to degrade the adhesives as a simulation for clinical use, exhibited less loss of bond strength compared to other filled resins. To determine whether bonding with ACP-containing orthodontic adhesives, designed to release ions at low pH, would be clinically durable is worthy of further investigation.

ACP-containing adhesive may be a great adjunct in the prevention of white spot lesions especially in cases where compliance is lacking. This study suggests that ACP-containing adhesive can provide similar bond strengths to at least one orthodontic adhesive that is already on the market and minimally meet the bond strength recommendations of Tavas and Watts.[12] Future studies, examining the efficacy of these adhesives in preventing white spot lesions, appear warranted.

CONCLUSIONS

- There was no significant difference in shear bond strength between Fuji Ortho LC and Aegis Ortho, both of which were significantly less than Transbond XT.
- The Weibull analysis showed the ACP-containing adhesive possessed a distribution of bond strengths that suggests its use may result in a greater tendency for early debonds. However, this should be factored with its positive claims, albeit not proven, of preventing white spots.
- Transbond XT and Fuji Ortho LC displayed a significantly different ARI score compared to Aegis Ortho; however, in all three groups, most of the adhesive was removed with the bracket.

ACKNOWLEDGMENTS

This project would not have been possible without the help of Dr Conlin and the Marquette University Oral Surgery Department in aiding with the collection of teeth. The authors are also grateful to the 3M/Unitek Corporation for their generous donations and extend thanks to Mary Modlinski for all her efforts in obtaining the materials needed to conduct this study.

REFERENCES

1. Basdra EK, Huber H, Komposch G. Fluoride released from orthodontic bonding agents alters the enamel surface and inhibits enamel demineralization in vitro. *Am J Orthod Dentofacial Orthop.* 1996;109:466–472.
2. Ogaard 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.
3. Glatz EGM, Featherstone JBD. Demineralization related to

orthodontic band and brackets—a clinical study. *Am J Orthod.* 1985;87:87.
4. Skrtic D, Hailer AW, Antonucci JM, Takagi S, Eanes ED. Quantitative assessment of the efficacy of amorphous calcium phosphate/methacrylate composites in remineralizing caries-like lesions artificially produced in bovine enamel. *J Dent Res.* 1996;75:1679–1686.
5. Skrtic D, Antonucci JM, Eanes ED. Improved properties of amorphous calcium phosphate fillers in remineralizing resin composites. *Dent Mater.* 1996;12:295–301.
6. Skrtic D, Antonucci JM, McDonough WG, Lui DW. Effect of chemical structure and composition of the resin phase on mechanical strength and vinyl conversion of amorphous calcium phosphate-based composites. *J Biomed Mater Res.* 2004;68A:763–772.
7. Daub J, Berzins DW, Linn BJ, Bradley TG. Bond strength of direct and indirect bonded brackets after thermocycling. *Angle Orthod.* 2006;76:295–300.
8. Northrup RG, Berzins DW, Bradley TG, Schuckit W. Shear bond strength comparison between two orthodontic adhesives and self ligating and conventional brackets. *Angle Orthod.* In press.
9. Benson PE, Shah AA, Millett DT, Dyer F, Parkin N, Vine RS. Fluorides, orthodontics and demineralization: a systematic review. *J Orthod.* 2005;32:102–114.
10. Fox NA, McCabe JF, Buckley JG. A critique of bond strength testing in orthodontics. *Br J Orthod.* 1994;21:33–43.
11. Linn BJ, Berzins DW, Dhuru VB, Bradley TG. A comparison of bond strength between direct- and indirect-bonding methods. *Angle Orthod.* 2006;76:289–294.
12. Tavas MA, Watts DC. A visible light activated direct bonding material: an in vitro comparative study. *Br J Orthod.* 1984;11:33–37.
13. Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod.* 1975;2:171–178.
14. Owens SE Jr, Miller BH. A comparison of shear bond strengths of three visible light-cured orthodontic adhesives. *Angle Orthod.* 2000;70:352–356.
15. Valente RM, de Rijk WG, Drummond JL, Evans CA. Etching conditions for resin-modified glass ionomer cement for orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 2002;121:516–520.
16. Rix D, Foley TF, Mamandras A. Comparison of bond strength of three adhesives: composite resin, hybrid GIC, and glass-filled GIC. *Am J Orthod Dentofacial Orthop.* 2001;119:36–42.
17. Bishara SE, VonWald L, Olsen ME, Laffoon JF. Effect of time on the shear bond strength of glass ionomer and composite orthodontic adhesives. *Am J Orthod Dentofacial Orthop.* 1999;116:616–620.
18. Dunn WJ. Shear bond strength of an amorphous calcium-phosphate-containing orthodontic resin cement. *Am J Orthod Dentofacial Orthop.* 2007;131:243–247.
19. Kawabata R, Hayakawa T, Kasai K. Modification of 4-META/MMA-TBB resin for safe debonding of orthodontic brackets—influence of the addition of degradable additives or fluoride compound. *Dent Mater J.* 2006;25:524–532.
20. Gaworski M, Weinstein M, Borislow AJ, Braitman LE. Decalcification and bond failure: a comparison of a glass ionomer and a composite resin bonding system in vivo. *Am J Orthod Dentofacial Orthop.* 1999;116:518–521.
21. Hitmi L, Muller C, Mujajic M, Attal JP. An 18-month clinical study of bond failures with resin-modified glass ionomer cement in orthodontic practice. *Am J Orthod Dentofacial Orthop.* 2001;120:406–415.
22. Summers A, Kao E, Gilmore J, Gunel E, Ngan P. Comparison of bond strength between a conventional resin adhesive and a resin-modified glass ionomer adhesive: an in vitro and in vivo study. *Am J Orthod Dentofacial Orthop.* 2004;126:200–206.
23. 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 a resin-reinforced glass ionomer cement: an in vitro and in vivo study. *Angle Orthod.* 1999;69:463–469.
24. Derks A, Katsaros C, Frencken JE, Van't Hof MA, Kuijpers-Jagtman AM. Caries-inhibiting effect of preventive measures during orthodontic treatment with fixed appliances. A systematic review. *Caries Res.* 2004;38:413–420.
25. O'Reilly MM, Featherstone JD. Demineralization and remineralization around orthodontic appliances: an in vivo study. *Am J Orthod Dentofacial Orthop.* 1987;92:33–40.
26. Geiger AM, Gorelick L, Gwinett AJ, Benson BJ. Reducing white spot lesions in orthodontic populations with fluoride rinsing. *Am J Orthod Dentofacial Orthop.* 1992;101:403–407.
27. Gorton J, Featherstone JD. In vivo inhibition of demineralization around orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 2003;123:10–14.
28. Trimpeneers LM, Dermaut LR. A clinical evaluation of the effectiveness of a fluoride-releasing visible light-activated bonding system to reduce demineralization around orthodontic brackets. *Am J Orthod Dentofacial Orthop.* 1996;110:218–222.