Original Article

Post-gel shrinkage, elastic modulus, and stress generated by orthodontic adhesives

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ABSTRACT

Objectives: To measure post-gel shrinkage, elastic modulus, and flexural strength of orthodontic adhesives and to predict shrinkage stress using finite element analysis (FEA).

Materials and Methods: The following 6 orthodontic adhesives were tested: Transbond XT (3M Unitek, Monrovia, Calif), Transbond Plus Color Change (3M Unitek), Greengloo (Ormco, Brea, Calif), Ortho Connect (GC America, Alsip, Ill), Trulock (RMO, Denver, Colo), GoTo (Reliance, Itasca, Ill). Post-gel shrinkage was measured using a biaxial strain gauge during light curing. Elastic modulus and flexural strength were measured with a 4-point bending test. Analysis of variance and Student-Newman-Keuls post hoc tests were used to compare the shrinkage, elastic modulus, and flexural strengths among the materials ($\alpha = .05$). Shrinkage stresses caused by the post-gel shrinkage and elastic modulus values were calculated using a cross-sectional FEA of a metallic bracket bonded to an incisor.

Results: Properties were highly different among the adhesives ($P \leq .0001$). Transbond XT (0.38 ± 0.09 percent volumetric contraction) and GoTo (0.42 ± 0.05 percent volumetric contraction) had the lowest post-gel shrinkage; Transbond Plus Color Change had the highest (0.84 ± 0.08 percent volumetric contraction). OrthoConnect (6.8 ± 0.6 gigapascals) had the lowest elastic modulus; GoTo (28.3 ± 3.1 gigapascals) had the highest. Trulock (64.1 ± 8.2 megapascals) had the lowest flexural strength; Greengloo (139.1 ± 20.7 megapascals) had the highest. FEA showed that the highest shrinkage stresses were generated with Transbond Plus Color Change and the lowest with OrthoConnect.

Conclusions: Post-gel shrinkage of orthodontic adhesives was comparable with restorative composites, which are known to create shrinkage stresses in restored teeth. FEA indicated that this shrinkage creates stresses in the adhesive and in the enamel around the brackets. (*Angle Orthod.* 2020;90:278–284.)

KEY WORDS: Post-gel shrinkage; Elastic modulus; Finite element analysis; Adhesive; Stress; Orthodontics

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Accepted: July 2019. Submitted: March 2019.
Published Online: September 23, 2019

 DOI: 10.2319/032719-233.1

INTRODUCTION

The advent of resin-based composites not only revolutionized the field of dentistry but also orthodontic practice.[1] With orthodontic composites (hereafter referred to as orthodontic adhesives because of the existing nomenclature in the discipline), clinicians could bond brackets directly to the tooth surface as opposed to relying on a band around the full circumference of the crown. However, this new, precarious position of the bracket on the facial surface came with its own problems. The bracket needed to be able to withstand the forces incurred during orthodontic treatment.[2] Thus, the propensity of brackets to "break," or lose their bond to the tooth and/or bracket base, is of particular relevance to orthodontic practice.

Clinically, bonded orthodontic brackets are subjected to masticatory loads as well as forces transferred by the wire being used to reposition the teeth.[2] In addition, the adhesive is subjected to internal stresses brought about by contraction during the polymerization process of the resin component.[3] Polymerization shrinkage of resin composites is able to generate stress levels capable of causing cuspal deflection of restored teeth and has been associated with the failure of adhesive bonds.[4] Polymerization of orthodontic adhesives may similarly affect the stability of bracket bonding or could stress the underlying enamel, but few studies considered polymerization shrinkage for orthodontic adhesives.[5,6] It is therefore important to understand shrinkage stresses that may occur in an orthodontic bracket-adhesive-enamel complex, as such stresses are in addition to aforementioned functional stresses incurred during orthodontic treatment and could further increase the risk of enamel damage during bracket debonding.

The purpose of this study was to evaluate how much orthodontic adhesives shrink during polymerization and how much shrinkage stress is potentially generated when the adhesives are used to bond a bracket to a tooth. Post-gel shrinkage was measured for six orthodontic adhesives using a strain gauge method. Shrinkage stresses do not only depend on the shrinkage but also involve mechanical properties (mainly elastic modulus), geometric factors, and the load and constraint conditions. Four-point bending tests were therefore carried out to determine the necessary mechanical properties of the orthodontic adhesives (elastic modulus and flexural strength), whereas finite element analysis was used to incorporate all relevant material, shape, and constraint factors for the calculation of stresses.

MATERIALS AND METHODS

The following six commonly used orthodontic adhesives were tested: Transbond XT Light Cure (3M Unitek, Monrovia, Calif), Transbond Plus Color Change (3M Unitek, St Paul, Minn), Greengloo (Ormco, Brea, Calif), Ortho Connect 2-in-1 primer and adhesive (GC America, Alsip, Ill), Trulock Light Activated Adhesive Paste (RMO, Denver, Colo), GoTo Adhesive Paste (Reliance, Itasca, Ill). The adhesives were selected by polling faculty clinicians in the Orthodontic Department of the University of Tennessee Health Science Center College of Dentistry.

Post-Gel Shrinkage Measurement

A biaxial strain gauge (CEA-06-032 WT-120; Micro Measurements Group, Raleigh, NC) was used to measure post-gel shrinkage.[7] Uncured adhesive specimens (approximately 1 mm thick, 3 mm diameter) were placed on the strain gauge (Figure 1A) and pressed down over the embedded sensor to ensure attachment. The strain gauge was integrated with a quarter bridge module (CompactDAQ system, National Instruments, Austin, Tex), which was connected to a desktop computer. The specimen was light cured from the top using an light emitting diode (LED) light source (DemiUltra, Kerr, Orange, Calif) for 20 seconds. The light tip was at about 3 mm distance from the strain gauge surface. The irradiance of the light-curing unit was 1234 mW/cm^2. The strain output of the gauge was acquired in real time during polymerization using a data acquisition program custom written in LabView (National Instruments). Strains were recorded for 10 minutes from the start of light curing. The value of the averaged two strain directions at 10 minutes was the linear post-gel shrinkage (α), which could be converted to percent volumetric shrinkage using $(3\,\alpha - 3\,\alpha^2 + \alpha^3) \times 100\%$. The sample size was 10 per adhesive.

Elastic Modulus and Flexural Strength Measurements

Bar-shaped specimens (2 mm $\times$ 2.5 mm $\times$ 25 mm) were fabricated from the orthodontic adhesives using a silicone mold. Specimens were light cured from the top and bottom sides, each for a total of 160 seconds. The specimens were smoothed using silicon carbide paper and stored away from light at room temperature for 24 hours. Width (W) and height (H) of the specimens were measured with a digital caliper at both ends and in the middle of the bar. These dimensions were averaged for each specimen. Elastic modulus and flexural strength were determined with a four-point bending test, where the distance between the two lower supports (L) was 20 mm and the distance between the upper supports was 10 mm (Figure 1B). The bars were loaded in a universal testing machine (Model 5567, Instron Corp., Norwood, Mass) at 0.5 mm/min until failure. The

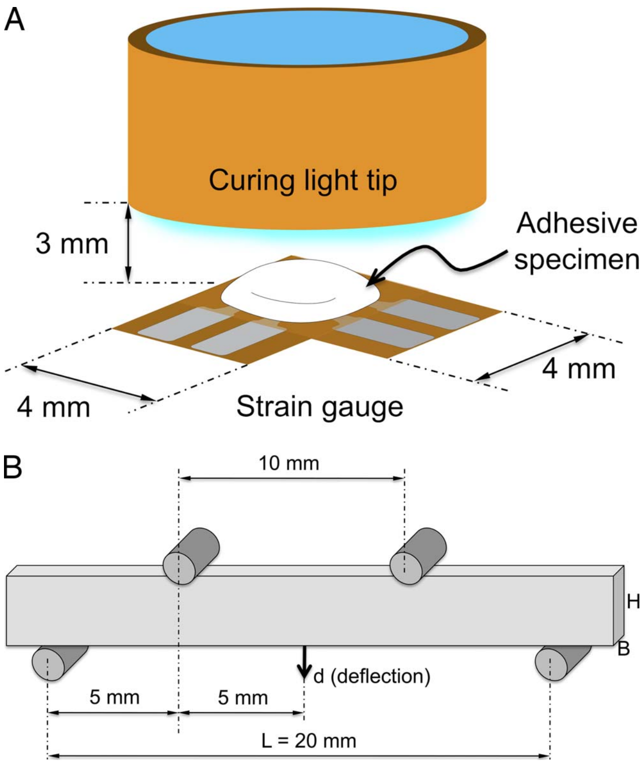

Figure 1. (A) Adhesive specimen placed on biaxial strain gauge and cured from the top. (B) Four-point bending bar configuration and dimensions.

applied load (F) and load at failure (F_{max}) were recorded with a 1 kN loadcell. The bending displacement (d) at the center of the bars was measured with a deflectometer (Model 3540-004M-ST Deflection Gage, Epsilon Technology Corp., Jackson, Wyo). Elastic modulus was calculated from the amount of center bending displacement using the following relationship: $(11\ F\ L^3)/(64\ W\ H^3\ d)$. Flexural strength, which is the maximum tensile strength found at the bottom of the bar specimen during a bending test, was calculated from $(3\ F_{max}\ L)/(4\ W\ H^2)$.[8] The sample size was 10 per adhesive.

Statistical Analysis

The experimental data were statistically analyzed using one-way analysis of variance followed by Student-Newman-Keuls post hoc test (significance level .05), and correlations between properties were tested using Pearson correlation.

Finite Element Analysis

For the calculation of shrinkage stresses, the measured shrinkage and elastic modulus values were applied in a finite element analysis of a stainless-steel bracket bonded to a maxillary central incisor. To create the finite element model, the cross-sectional outlines of an incisor and bracket were traced and imported into a finite element program (Marc/Mentat, MSC Software, Palo Alto, Calif). An element mesh was created using plane strain quadrilateral elements, with the bracket bonded to the facial tooth surface via a 150 μm thick adhesive layer (Figure 2). The crown mesh was more refined because all shrinkage stresses were confined to the crown. Each tissue/material was assigned material properties obtained from the literature[9,10] or provided by the bracket manufacturer (Table 1). All materials were assumed to be isotropic and the properties to be homogeneous and linear elastic. Polymerization shrinkage for each orthodontic adhesive was modeled by inducing volume contraction corresponding to the experimentally measured post-gel shrinkage. Only in-plane shrinkage was allowed to avoid artifacts from the thickness restraints posed by plane strain conditions that would otherwise induce high out-of-plane shrinkage stresses. The tooth model was kept in place by fixing the bottom and lateral bone edges. First (or maximum) principal stresses were calculated and their distributions visualized using a linear color scale. Principal stresses represent all stress components, including all shear stresses; the first principal stress was the highest value of the three principal stresses. The mean of the 20 highest adhesive and enamel stress values (collected in all four integration points of adhesive and enamel elements) were used to compare the models. Using the mean of the 20 highest stress values instead of the maximum stress value avoided skewing the outcomes by singular stress peaks and restricted the selection to enamel areas around the brackets that experienced stresses.

RESULTS

Significant differences in post-gel shrinkage, elastic modulus, and flexural strength were found among the six orthodontic adhesives ($P = .0001$ for all properties). Table 2 lists the results for post-gel shrinkage expressed as percent volumetric contraction (%volume), elastic modulus in gigapascals (GPa), and flexural strength in megapascals (MPa).

Most shrinkage took place in the first minute, as can be seen in Figure 3, which shows the mean shrinkage strain curve for each orthodontic adhesive. Negative strain values indicate shrinkage, and positive values indicate expansion. Temperature increase as a result of the exotherm reaction and irradiation caused an initial expansion. The shrinkage curves were practically level at 10 minutes. Post-gel shrinkage values at 10 minutes (Table 2) showed that Transbond XT (0.38 $\pm$ 0.09 %volume) and GoTo (0.42 $\pm$ 0.05 %volume) had

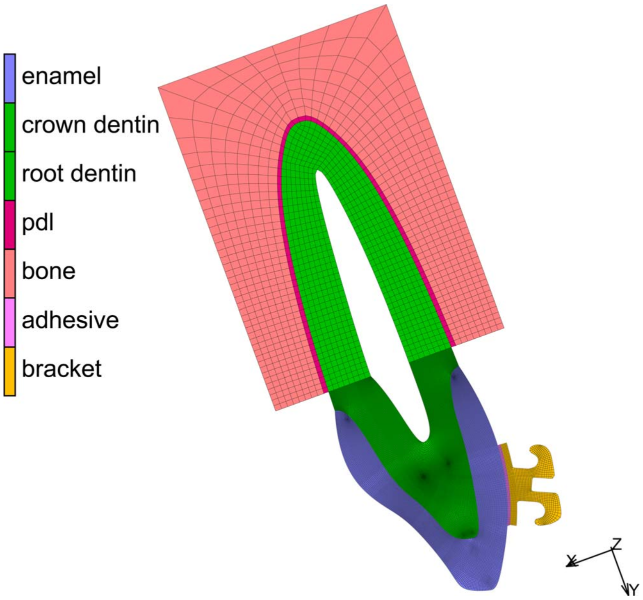

Figure 2. Cross-sectional finite element model of incisor with stainless steel bracket.

the lowest post-gel shrinkage values, whereas Transbond Plus Color Change had the highest (0.84 ± 0.08 %volume).

The elastic modulus of the tested adhesives ranged from 3.4 ± 0.3 GPa (OrthoConnect) to 14.2 ± 1.6 GPa (GoTo). Flexural strength ranged from 64.1 ± 8.2 MPa (Trulock) to 139.1 ± 20.7 MPa (Greengloo) and did not show the same ranking as elastic modulus. There was no correlation among the three mechanical properties (Pearson correlation coefficients ranged from –0.1965 to 0.0849).

Maximum stress levels (mean of top-20 first principal stress values) in enamel and orthodontic adhesive calculated in the finite element models are summarized in Table 2, showing the highest stress levels with Transbond Plus Color Change and the lowest with OrthoConnect. Maximum stresses in the adhesive were considerably lower than the flexural strength. The stress table indicates a perfect correlation of the maximum shrinkage stress levels between orthodontic adhesives and enamel (coefficient of correlation 0.9977). The stress levels in enamel and orthodontic adhesives showed a strong correlation with post-gel shrinkage (correlation coefficients 0.7108 and 0.6678, respectively) and with elastic modulus (correlation coefficients 0.6130 and 0.6584, respectively). The stress distributions are shown in Figure 4, where each material (enamel, adhesive, and bracket) has its own stress scale because stress values depend on elastic modulus and the relevance of a stress value depends on the strength of its material. Relatively uniform stress distributions were found across the adhesive layer,

Table 1. Material Properties Used in the Finite Element Analysis

Material	Elastic Modulus (GPa[a])	Poisson's Ratio
Enamel	84.1	0.30
Dentin	18.3	0.23
Periodontal ligament	0.05	0.45
Bone	13.7	0.33
Orthodontic adhesive	see Table 2	0.30
Stainless steel (bracket)	197.0	0.27

[a] GPa indicates gigapascals.

whereas prominent stress concentrations were seen in the enamel at the bracket edges.

DISCUSSION

The results of this study confirmed substantial polymerization shrinkage during curing of all tested orthodontic adhesives and subsequent stress analysis indicated that this polymerization shrinkage had the potential of considerable residual stresses in the adhesive layer and in the enamel surrounding a bonded metallic bracket. In orthodontics, polymerization shrinkage and shrinkage stress have received little attention. Shrinkage values of orthodontic adhesive resins were evaluated in a few studies.[5,6,11] The authors in those studies pointed to high C-factors (which assess shrinkage stress based on bonded/unbonded surface ratio) of the thin adhesive layer between rigid enamel and bracket to support their assumption that bonding orthodontic brackets should have high shrinkage stresses. However, the C-factor concept is inadequate for predicting shrinkage stresses as it does not take material properties, actual geometry, or substrates into account and disregards stress distributions and concentrations. In the case of orthodontic brackets, for example, the distance between bracket and tooth surface is not fixed but is free to adapt and settle (as if "floating") during polymerization shrinkage. The current study used finite element analysis to calculate stresses in the tooth-bracket system, which allowed us to take the material properties of adhesives and substrates into account as well as the geometric factors.

This study used the strain gauge method to measure shrinkage. Strain curves recorded during polymerization offered an insight into the different characteristics of the adhesive materials. Steeper shrinkage curves, such as for Transbond Plus Color Change, indicated a faster reaction, which was associated with more post-gel shrinkage because a faster cure allows less stress relief. Note that curing lights with higher irradiance could further increase polymerization rate and thus post-gel shrinkage but, in this study, the same light source and light tip position was used for all measurements to maintain comparable outcomes. The shal-

Table 2. Post-Gel Shrinkage, Elastic Modulus, and Flexural Strength (Mean ± Standard Deviation) and Shrinkage Stresses of Tested Orthodontic Adhesives[a]

Orthodontic Adhesives	Post-Gel Shrinkage (%volume)	Elastic Modulus (GPa)	Flexural Strength (MPa)	First Principal Stress (Mean of Top 20 Stresses; MPa)	
				Enamel	Adhesive
Ortho Connect (GC America, Alsip, Ill)	0.53 ± 0.12[c,d]	3.4 ± 0.3[a]	102.3 ± 16.2[b,c]	2.85	8.94
Trulock (RMO, Denver, Colo)	0.58 ± 0.10[d]	9.8 ± 1.3[b]	64.1 ± 8.2[a]	9.56	28.06
Transbond XT (3M Unitek, Monrovia, Calif)	0.38 ± 0.09[a]	11.1 ± 0.9[c]	114.1 ± 10.2[c]	6.97	20.36
Transbond Plus Color Change (3M Unitek)	0.84 ± 0.08[e]	11.3 ± 1.1[c]	101.4 ± 23.2[b,c]	16.02	46.79
Greengloo (Ormco, Brea, Calif)	0.48 ± 0.06[b,c]	12.3 ± 1.1[d]	139.1 ± 20.7[d]	9.92	28.93
GoTo (Reliance, Itasca, Ill)	0.42 ± 0.05[a,b]	14.2 ± 1.6[e]	86.3 ± 19.1[b]	9.96	28.96

[a] Different superscript letters indicate significant difference among the adhesives within each property (analysis of variance and Student-Newman-Keuls post hoc, $\alpha = .05$). %volume indicates percent volumetric contraction; GPa, gigapascals; MPa, megapascals.

lower curves, such as Transbond XT and GoTo, indicated slower polymerization and resulted in the lowest post-gel shrinkage. The shrinkage values found in this study were much lower than the values reported in the studies mentioned previously because the strain gauge measured only the portion of contraction that caused stress (ie, post-gel shrinkage). Usually total contraction[5] or constrained contraction[6] is reported as the shrinkage value. However, not all of that contraction causes stress, as it was shown that only post-gel shrinkage had a correlation with shrinkage stresses.[12,13] The correlation between post-gel shrinkage and shrinkage stress was reconfirmed in this study for orthodontic brackets.

The current study also confirmed the correlation between the elastic modulus and shrinkage stresses. The elastic modulus of the orthodontic adhesives is an essential factor in stress development, whereas flexural strength gives stress values perspective (eg, a high stress in a strong material can be less critical than a low stress in a weak material) and has been associated with bond strength.[14] Both properties were determined with four-point bending tests. The elastic modulus values were substantially higher than some reported in the literature. This study found an elastic modulus of 11.1 GPa for Transbond XT, whereas 4.7 GPa[11] and 8.3 GPa[14] were reported in the literature. On the other hand, a Food and Drug Administration document listed 16.5 GPa manufacturer value for Transbond XT (https://www.accessdata.fda.gov/cdrh_docs/pdf16/K160782.pdf). A dedicated deflectometer was used in this study that was more accurate for measuring small displacements than the internal extensometer of the testing machine. Flexural strength does not involve measurement of the displacement, and the 114.1 MPa found for Transbond XT in this study was closer to literature values (152.7 MPa[11] and 145.3 MPa[14]) and the manufacturer's 123.0 MPa. Comparing the post-gel shrinkage with elastic modulus and flexural strength showed that there was no correlation among these properties. This observation reemphasized that shrinkage stresses should never be extrapolated from single properties (such as shrinkage) because material properties are independent of each other and stress is determined by a combination of properties and conditions.

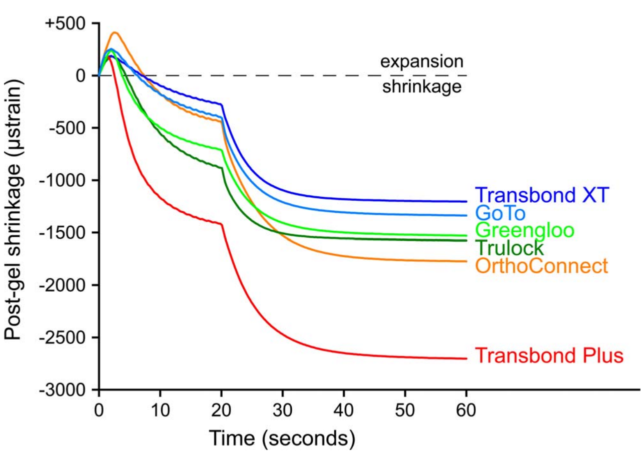

Figure 3. Mean strain curve for each of the six orthodontic adhesives during the first 60 seconds after start of polymerization, showing initial expansion (positive strains) followed by shrinkage (negative strains).

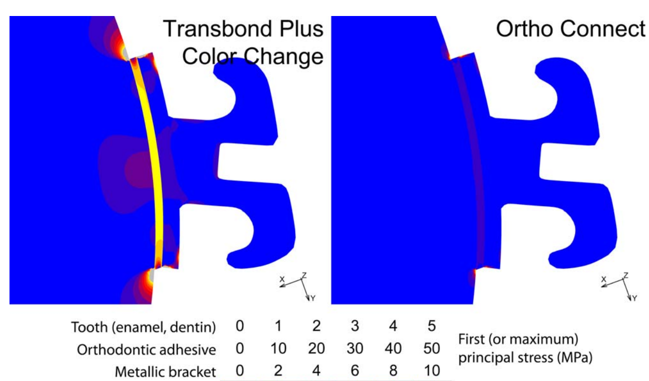

Figure 4. Shrinkage stress distributions of the first (maximum) principal stress generated by polymerization of two orthodontic adhesives that represented the highest and lowest shrinkage stress levels of the six materials evaluated.

Using the experimentally determined properties of the orthodontic adhesives and combining them with the geometric configuration and bonding conditions of a metallic orthodontic bracket in the finite element analysis demonstrated the resulting stress distributions. Finite element analysis has become an important tool in dental research because it allows the consideration of experimentally determined properties within a larger complex of properties and configurations for determination of stress conditions. In this study, the orthotropic property of enamel (ie, higher elastic modulus in the direction of enamel rods) was disregarded, which may have slightly affected the stress distributions in the enamel substrate close to the bracket. In addition, a cross-sectional model was used that could not account for geometrical variation out of the plane because the employed plane strain conditions assumes infinite tooth and bracket widths. Nevertheless, the model can be considered a reasonable representation of stress distributions in adhesives and enamel at a bracket cross-section, allowing several relevant observations about the nature and consequences of shrinkage in orthodontic adhesives.

First, despite brackets “floating” over the tooth surface, substantial polymerization shrinkage stresses can develop in orthodontic adhesives, where the highest values are found at the enamel and bracket interfaces. Given the good clinical experience with each of the six tested adhesives, the stresses were obviously not high enough to break the bonds or exceed the strength of the adhesive materials (compare maximum stresses with the flexural strength values). Yet, these shrinkage stresses are in addition to any functional and masticatory stresses transmitted through the bracket and therefore the presence of shrinkage stresses is likely to increase the risk of premature bracket debonding. This study tested the post-gel shrinkage for specimens that were light cured directly from above. Clinically, adhesives would be partly covered by a bracket, which likely reduces the polymerization rate and thus post-gel shrinkage. In addition, polymerization shrinkage was only tested immediately after curing. In resin composites, hygroscopic expansion was shown to counteract shrinkage and thus reduces shrinkage stresses over time.[4,15] Under clinical conditions, the same stress reduction may happen in orthodontic adhesives.

Second, orthodontic adhesive shrinkage causes stresses in the enamel around the periphery of a bracket. This is the same area where many patients develop white spot lesions after orthodontic treatment.[16,17] White spot lesions signify decreased calcification of the enamel from cariogenic acid production.[18] The peripheral bracket areas are particularly susceptible to bacterial plaque accumulations because they are difficult to clean.[19] Demineralized enamel is structurally compromised and is more easily damaged.[18] If such a compromised area is subjected to an additional shrinkage stress, masticatory forces or the force applied during bracket removal are more prone to result in enamel fracture.

Last, it is notable that the post-gel shrinkage values of the orthodontic adhesives were comparable with those of restorative composites, which are widely considered to be a serious issue for dental restorations.[4,13] The clinical performance of an orthodontic adhesive, however, cannot be characterized by single properties. The current study can therefore not recommend which of the six tested adhesives is best for orthodontic use. Nevertheless, it seems highly relevant for clinicians to be aware of the potential stress state around brackets and how various adhesives may affect the level of those stresses. Such information can help in the material selection and techniques to alleviate associated risks of premature debonding or enamel damage during bracket removal.

CONCLUSIONS

- Post-gel shrinkage, elastic modulus, and flexural strength varied greatly among the tested orthodontic adhesives.
- Finite element analysis revealed that the post-gel shrinkage and elastic modulus of orthodontic adhesives correlated with maximum shrinkage stresses in the adhesive and enamel, where the highest enamel stresses were located around the periphery of the bracket.

ACKNOWLEDGMENTS

Supported by the University of Tennessee Health Science Center College of Dentistry Alumni Endowment Fund and the Tennessee Dental Association Foundation. The authors thank GC Orthodontics, Rocky Mountain Orthodontics, 3M Unitek, Ormco, and Reliance Orthodontics for donating the orthodontic adhesives.

REFERENCES

1. Sharma S, Tandon P, Nagar A, Singh G, Singh A, Chugh VK. A comparison of shear bond strength of orthodontic brackets bonded with four different orthodontic adhesives. *J Orthod Sci*. 2014:3;29–33.
2. Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod*. 1975;2:171–178.
3. Bowen RL. Properties of a silica-reinforced polymer for dental restorations. *J Am Dent Assoc*. 1963;66:57–64.
4. Tantbirojn D, Pfeifer CS, Braga RR, Versluis A. Do low-shrink composites reduce polymerization shrinkage effects? *J Dent Res*. 2011;90:596–601.

5. Bang H-C, Lim B-S, Yoon T-H, Lee Y-K, Kim C-W. Effect of plasma arc curing on polymerization shrinkage of orthodontic adhesive resins. *J Oral Rehabil*. 2004;31:803–810.
6. Sener Y, Uysal T, Basciftci FA, Demir A, Botsali MS. Conventional and high-intensity halogen light effects on polymerization shrinkage of orthodontic adhesives. *Angle Orthod*. 2006;76:677–681.
7. Sakaguchi RL, Versluis A, Douglas WH. Analysis of strain gage method for measurement of post-gel shrinkage in resin composites. *Dent Mater*. 1997;13:233–239.
8. Gere JM, Timoshenko SP. *Mechanics of Materials*. 3rd ed. Boston, MA: PWS Publishing Company; 1990.
9. Carter DR, Hayes WC. The compressive behavior of bone as a two-phase porous structure. *J Bone Joint Surg Am*. 1977;59:954–962.
10. Craig RG, Powers JM. *Restorative Dental Materials*. St. Louis, MO: Mosby; 2002.
11. Gama ACS, Moraes AGV, Yamasaki LC, Loguercio AD, Carvalho CN, Bauer J. Properties of composite materials used for bracket bonding. *Braz Dent J*. 2013;24:279–283.
12. Boaro LCC, Gonçalves F, Guimarães TC, Ferracane JL, Versluis A, Braga RR. Polymerization stress, shrinkage and elastic modulus of current low-shrinkage restorative composites. *Dent Mater*. 2010;26:1144–1150.
13. Enochs T, Hill AE, Worley CE, Veríssimo C, Tantbirojn D, Versluis A. Cuspal flexure of composite-restored typodont teeth and correlation with polymerization shrinkage values. *Dent Mater*. 2018;34:152–160.
14. Li J. Effect of flexural strength of orthodontic resin cement on bond strength of metal brackets to enamel surfaces. *Eur J Orthod*. 2011;33:167–173.
15. Suiter EA, Watson LE, Tantbirojn D, Lou JSB, Versluis A. Effective expansion: balance between shrinkage and hygroscopic expansion. *J Dent Res*. 2016;95:543–549.
16. Gorelick L, Geiger AM, Gwinnett AJ. Incidence of white spot formation after bonding and banding. *Am J Orthod Dentofacial Orthop*. 1982;81:93–98.
17. Tüfekçi E, Merrill TE, Pintado MR, Beyer JP, Brantley WA. Enamel loss associated with orthodontic adhesive removal on teeth with white spot lesions: an in vitro study. *Am J Orthod Dentofacial Orthop*. 2004;125:733–739.
18. Mayne RJ, Cochrane NJ, Cai F, Woods MG, Reynolds EC. In-vitro study of the effect of casein phosphopeptide amorphous calcium fluoride phosphate on iatrogenic damage to enamel during orthodontic adhesive removal. *Am J Orthod Dentofacial Orthop*. 2011;139:e543–e551.
19. O'Reilly MM, Featherstone JDB. Demineralization and remineralization around orthodontic appliances: an in vivo study. *Am J Orthod Dentofacial Orthop*. 1987;92:33–40.