Original Article

Effect of Disinfecting Solutions on the Mechanical Properties of Orthodontic Elastomeric Ligatures

Maylani B. Evangelista[a]; David W. Berzins[b]; Peter Monaghan[c]

ABSTRACT

Objective: To assess the effect extended exposure to disinfectant solutions has on the tensile load at failure and glass transition temperature (T_g) of orthodontic elastomeric ligatures.

Materials and Methods: Elastomeric ligatures from three manufacturers: Rocky Mountain Orthodontics (RMO, Denver, Colo), American Orthodontics (AO, Sheboygan, Wis), and 3M Unitek (3M, Monrovia, Calif) were exposed to two disinfectant solutions, Vital Defense-D and Cidexplus, for up to 28 days. Unexposed ligatures were also tested. Tensile load at failure was determined by stretching the ligatures using a universal testing machine until they fractured. Glass transition temperature was determined using differential scanning calorimetry (DSC).

Results: For tensile load at failure and glass transition temperature of the ligatures, significant differences were observed among the different manufacturers and exposure times. Type of disinfectant solution was a significant factor with T_g, but not with failure load. The 3M ligatures had the highest tensile load at failure and most positive T_g followed by AO and RMO, respectively. Compared to unexposed ligatures, strength significantly decreased after one hour of disinfectant exposure. Glass transition temperature was also significantly affected with extended disinfectant exposure, but the different disinfectants changed T_g in opposite directions.

Conclusion: Exposure of elastomeric ligatures to disinfectant solution for one hour or more decreases their strength.

KEY WORDS: Ligatures; Strength; Disinfectant; DSC

INTRODUCTION

Polyurethane elastomers are used in orthodontics in the form of ligatures and chains or modules.[1] Numerous studies have been conducted to evaluate their strength, in terms of force delivery and rate of force decay in various environments and different testing conditions.[2–10] Factors such as tooth movement, temperature changes, pH variations, oral fluoride rinses, salivary enzymes, and masticatory forces have all been associated with the deformation, force degradation, and relaxation behavior of these elastomers. It has been found that a force loss of about 50% to 70% occurs in the first 24 hours followed by a steady decline over 3 to 4 weeks.[11]

Elastomeric ligatures are one of the important components in orthodontic treatment because they tie the archwire, which generates forces needed for tooth movement, to the brackets. The ease in application and cost efficiency make them more popularly used than other forms of ligation (eg, wire ligatures, self-ligating brackets). Unused portions of elastomeric ligatures are sterilized by cold sterilization methods as recommended by the Centers for Disease Control and Prevention (CDC) and the US Department of Labor Occupational Safety and Health Administration (OSHA) for instruments that cannot withstand heat.[12] Manufacturers of disinfectant solutions have minimum contact time and temperature requirements to ensure the effectiveness of antibacterial action, but they have not expressed any maximum contact time of exposure to the solutions. It is not unusual for an orthodontic clinic to be operating less than five days a week. Often, due to clinic operating schedules, these ligatures are immersed for longer, continuous hours, even days,

[a]Graduate Student (MS), Dental Biomaterials, Marquette University, Milwaukee, Wis.

[b]Assistant Professor, Dental Biomaterials, Marquette University, Milwaukee, Wis.

[c]Research Scientist and Private Practice, Evanston, Ill.

Corresponding author: David W. Berzins, PhD, Dental Biomaterials, Marquette University, 113A Wehr Physics, PO Box 1881, Milwaukee, WI 53201-1881 (e-mail: david.berzins@marquette.edu)

Accepted: July 2006. Submitted: May 2006.

before being removed and stored for future clinical use.

A limited number of studies testing the effect of antibacterial solutions on orthodontic elastomers appear in the literature.[2,3] Mayberry et al measured the force required to stretch three brands of modules a prescribed distance after 20 cyclic exposures of 10 minutes to a 0.5% glutaraldehyde solution, in addition to testing after other disinfection procedures. They found a slight weakening of the modules. Jeffries and von Fraunhofer examined the tensile force to fail of six elastomeric chains exposed to two brands of 2% alkaline glutaraldehyde solution for 30 minutes, 10 hours, and 144 hours. Compared to as-received material, they found a significant decrease in failure load in four of the chains after exposure to one brand for 10 hours. At 144 hours, all materials were significantly decreased in strength. Curiously, the other solution did not affect the failure load of the chains. In general, these studies show that exposure to disinfectant solution may result in a decrease in tensile strength and force delivery. However, prolonged, continuous exposure to antibacterial agents and its effect on the tensile strength of elastomeric ligatures has not been investigated. For orthodontic ligatures, maintenance of force delivery is needed to sustain full engagement of archwires in the bracket slot.[1]

In this investigation, short to medium exposure of elastomeric ligatures to disinfectant solution and its effect on strength was studied. A companion study was then performed to determine if any loss in strength was related to an alteration of the elastomeric ligature structure. This was accomplished by measuring the glass transition temperature (T_g), a property which is specific to the material and is related to its molecular structure, and therefore its rigidity and force delivery.[13–15] The glass transition represents the range of temperatures at which a material goes from a more rigid, glassy state to a flexible, rubbery state. In general, the higher the T_g, the more rigid the polymer. Conversely, flexible polymers, typically exhibit low T_g values. Renick et al[15] showed orthodontic elastomeric modules have T_g values of approximately -20°C to -50°C, which follows that they are very flexible at oral temperatures. No known studies have been published regarding the effect of antibacterial agents on the glass transition temperature of elastomeric ligatures.

MATERIALS AND METHODS

Gray elastomeric ligatures were obtained from three manufacturers: American Orthodontics (AO; Sheboygan, Wis), Rocky Mountain Orthodontics (RMO; Denver, Colo), and 3M Unitek (3M; Monrovia, Calif). These ligatures were stored at room temperature (22 ± 2°C) and unexposed to light in two disinfectant solutions for up to 28 days. The two disinfectant solutions used were: (1) Vital Defense-D (Vital Defense Company, Denver, Colo), which is a 9% o-phenylphenol and 1% o-benzyl-p-chlorophenol and (2) Cidexplus (Advanced Sterilization Products, Irvine, Calif), which is a 3.4% glutaraldehyde solution. The minimum contact time for Vital Defense-D is 10 minutes at 20°C for disinfection while that of Cidexplus is 20 minutes at 25°C for disinfection and 10 hours for sterilization.

Tensile load at failure testing

Ten ligatures from each manufacturer were stored in each disinfectant solution with exposure times of 10 minutes, 1 hour, 8 hours, 48 hours, 7 days, 14 days, and 28 days. In addition, ligatures unexposed to disinfectant solution were tested. These serve as a control and represent the common clinical situation of using the ligatures as received from the manufacturer. All control ligatures were stored dry at room temperature (22 ± 2°C), unexposed to light. Mechanical testing was performed by placing a specimen in a custom-made jig comprised of two metal pins attached respectively to the fixed and movable crossheads of a universal testing machine (Instron Corporation, Canton, Mass). Each ligature was loaded in tension at a crosshead speed of 100 mm/min until fracture occurred.[16] Maximum tensile load was recorded in Newtons. The tensile load at failure was used as an analog to the clinical situation of ligature breakage during tie-in.

Glass transition temperature testing

The glass transition temperature of the elastomeric ligatures was determined using a differential scanning calorimeter, or DSC (DSC822e, Mettler-Toledo Inc, Columbus, Ohio). A differential scanning calorimeter measures the heat flow of a sample over a range of temperatures, with the glass transition representing the range of temperatures at which a material goes from a more rigid, glassy state to a flexible, rubbery state. Correspondingly, there is a change in the heat capacity of the material that is able to be detected in the DSC thermogram. For each exposure condition, a single ligature was placed in an aluminum pan, and an empty aluminum pan served as the inert reference material for each DSC analysis. DSC thermograms were generated over a temperature range of -120°C to 150°C using a heating rate of 10°C/min. The DSC manufacturer's software was used to determine the glass transition temperature, typically presented as the midpoint of the glass transition temperature range. From each manufacturer, seven ligatures that had not been exposed to disinfectant were initially tested. Sim-

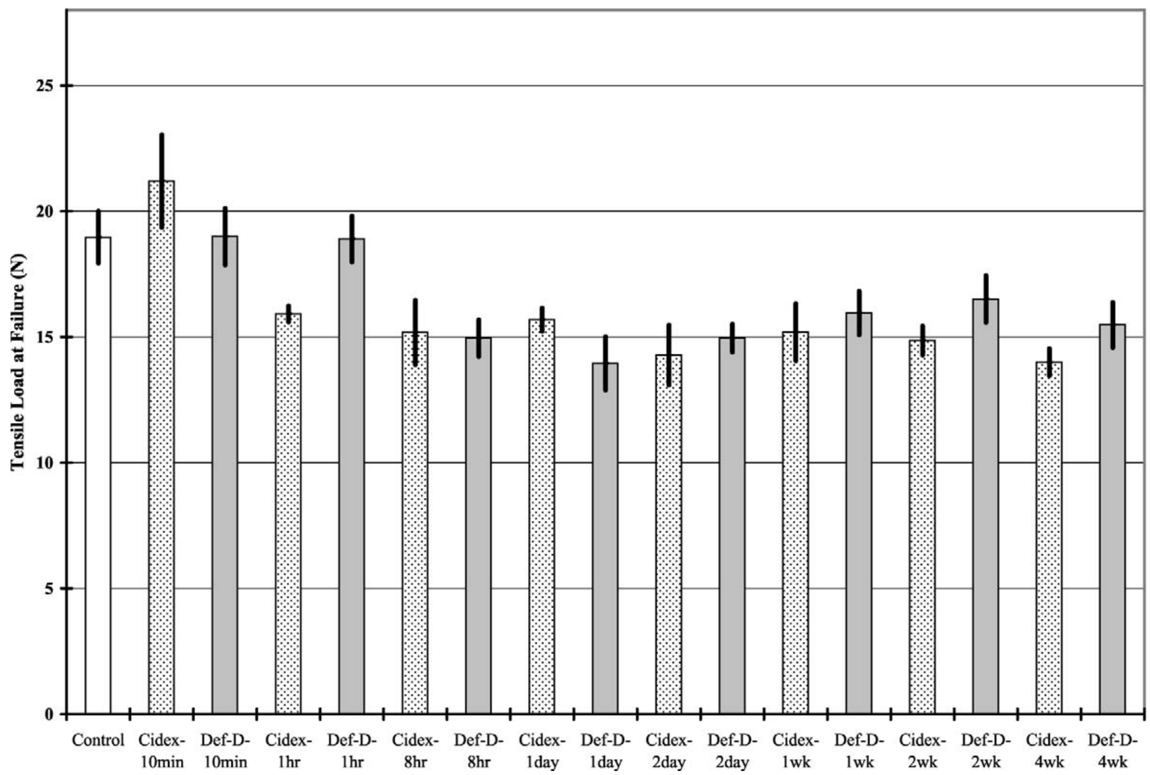

Figure 1. Tensile load at failure of ligatures from American Orthodontics exposed to Cidexplus and Vital Defense-D. The error bars represent standard deviation.

ilar to the storage described above, these control ligatures were stored dry at room temperature (22 ± 2°C), unexposed to light. Since longer exposure to disinfectant solution was expected to result in the greatest change in T_g, DSC testing was started on specimens ($n = 7$)[15] with the maximum exposure of 28 days and then progressed to lesser exposure times until a significant difference was no longer observed between exposed and unexposed specimens for any given ligature-disinfectant combination.

Statistical analysis

The data were tested for normality with Shapiro-Wilk's test followed by three-way analysis of variance (ANOVA) to find significant differences between ligature manufacturer, disinfectant solution, and exposure time (SPSS Inc, Chicago, Ill). A post hoc Tukey-Kramer HSD test was used to determine significant differences between manufacturers. A post-hoc Dunnett's test was used to determine significant differences with respect to disinfectant solution and time of immersion against the unexposed specimens. A level of confidence of 95% was used for all statistical calculations.

RESULTS

The results for the tensile load at failure testing are shown in Figures 1–3. Three-way ANOVA showed significant differences ($P < .0001$) within manufacturer and time of exposure but not between disinfectants ($P = .12$). Significant interactions ($P < .0001$) were observed between manufacturer and time and between disinfectant and time. A post hoc Tukey-Kramer HSD test showed that the failure loads of ligatures from all three companies were significantly different ($P < .0001$) from each other, with the ligatures from 3M exhibiting the greatest tensile load at failure and those from RMO, the least. In comparison to unexposed specimens, Dunnett's test showed that significantly ($P < .05$) lower failure loads were found in ligatures exposed to disinfectant for 1 hour or longer, with the trend of greater significance at longer exposure times.

A typical DSC thermogram for a ligature displaying the computer software determination of glass transition temperature is shown in Figure 4. The results for the glass transition temperature testing are displayed in Table 1. The three-way ANOVA showed significant differences within manufacturer ($P < .0001$), disinfectant ($P < .0001$), and time exposure ($P < .05$).

There was a significant interaction ($P < .05$) between disinfectant and time with regard to glass transition temperature. A post hoc Tukey-Kramer HSD test showed the T_g values of ligatures from all three companies were significantly different ($P < .005$) from each other, with the ligatures from 3M exhibiting the

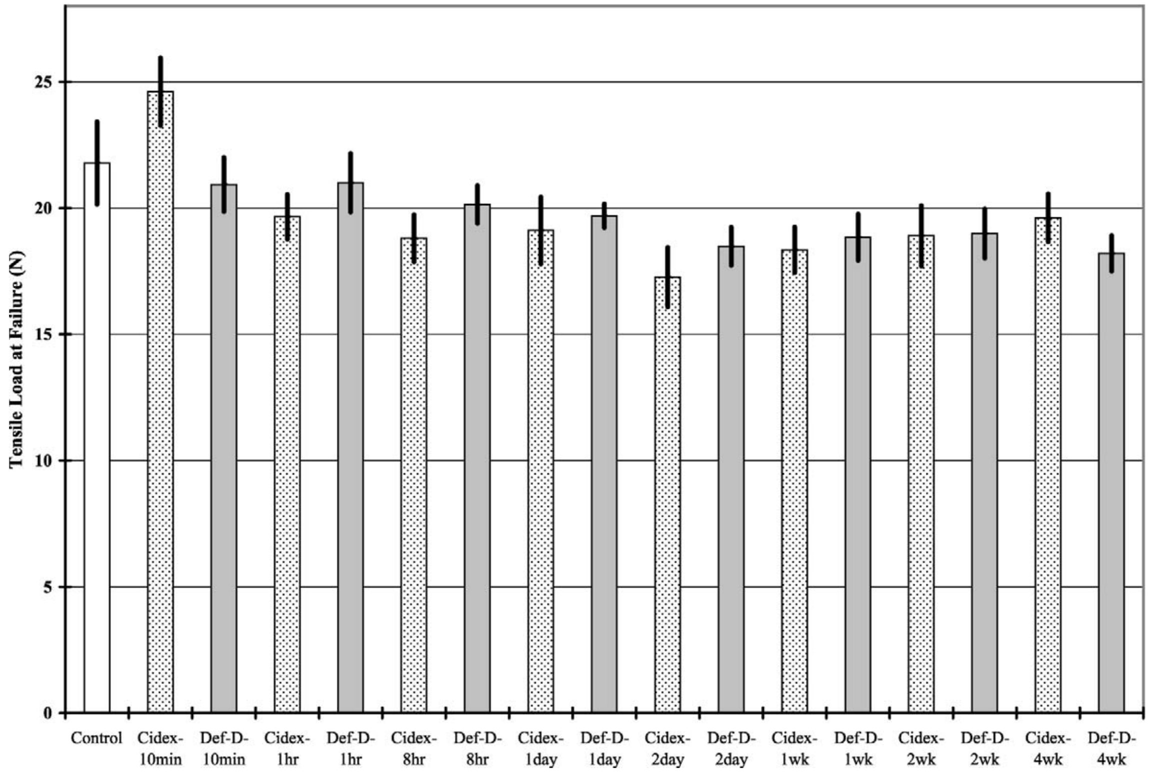

Figure 2. Tensile load at failure of ligatures from 3M Unitek exposed to Cidexplus and Vital Defense-D. The error bars represent standard deviation.

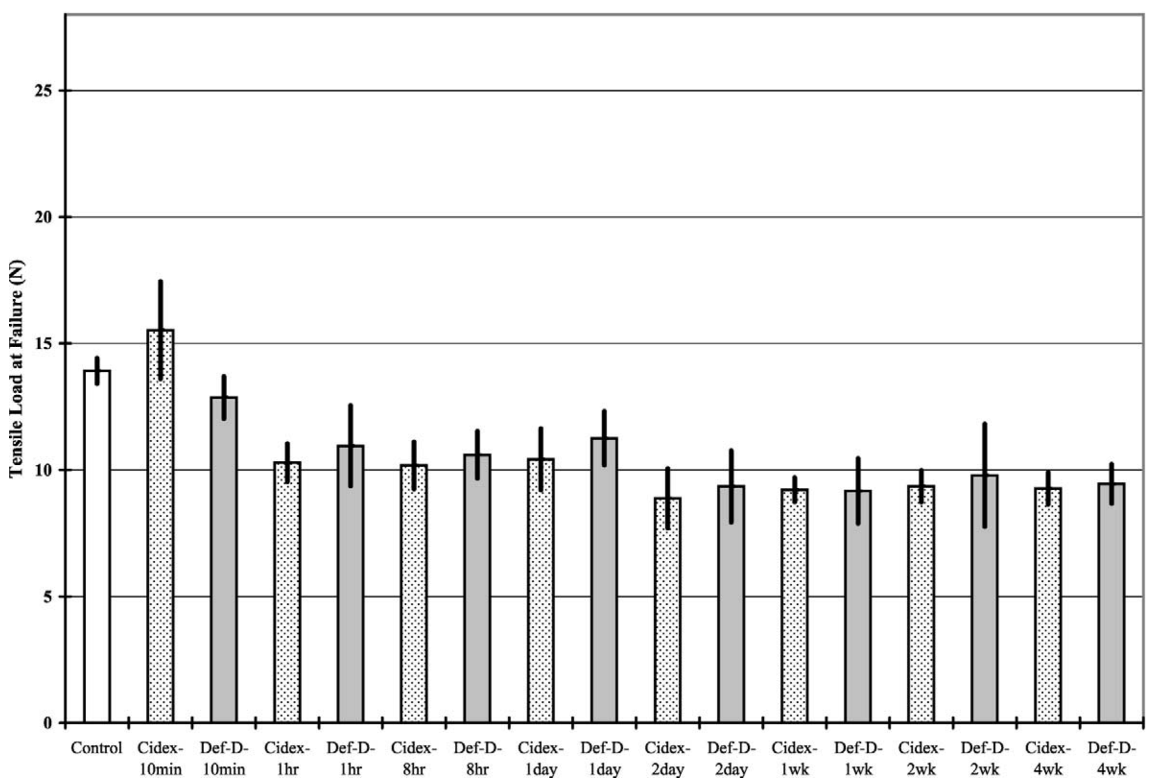

Figure 3. Tensile load at failure of ligatures from Rocky Mountain Orthodontics exposed to Cidexplus and Vital Defense-D. The error bars represent standard deviation.

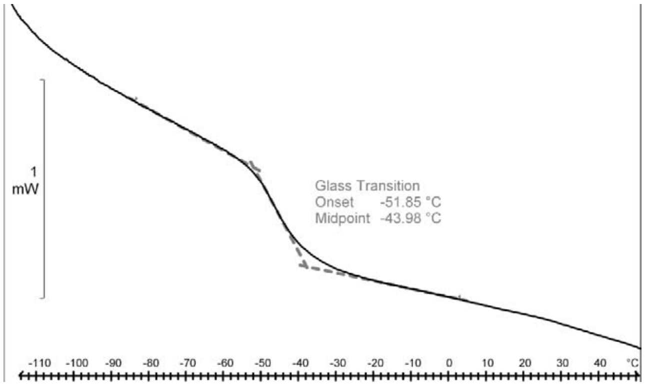

Figure 4. Representative differential scanning calorimetry (DSC) thermogram for a ligature illustrating the glass transition. The glass transition temperature (T_g) is given as the midpoint of a line connecting the two baselines (dotted lines drawn with the DSC software program).

greatest T_g and those from RMO, the most negative. For ligatures from AO and 3M, the type of disinfectant used affected the glass transition temperature differently. Compared to the T_g of unexposed specimens, the T_g of the AO and 3M ligatures increased or became more positive with exposure to Vital Defense-D, and it decreased or became more negative with exposure to Cidexplus. This trend was consistent and continued as exposure time increased from 14 to 28 days. The glass transition temperature of the ligatures from RMO, however, became more positive with exposure to either disinfecting solution. With the exception of the AO ligatures exposed to Cidexplus for 14 days, all exposure groups exhibited significantly ($P < .05$; Dunnett's post hoc test) different T_g values from the respective unexposed ligatures.

DISCUSSION

The tensile load at failure and glass transition temperature values of the unexposed ligatures from each manufacturer were significantly different from each other. This may be attributed to small differences in production despite the fact that most of the orthodontic elastomers currently available share similar fabrication methods. Some factors identified previously include: processing variations in manufacturing techniques involving cutting or injection-molding of the raw material, effects induced from various additives incorporated in the final product, and different morphologic or dimensional characteristics.[17] Overall, the tensile load at failure and glass transition temperature rankings among the manufacturers are in agreement. Both tensile load at failure and T_g followed the order: 3M > AO > RMO. A higher value of glass transition temperature (more positive) indicates a more rigid polymer, by either the presence of more cross-linked types of covalent bonding or larger side chains causing steric interferences[15]; this, therefore, follows that a stronger material has a higher tensile strength value.

Polyurethanes are not inert materials and instead are subject to water absorption and degradation with prolonged contact with enzymes, water, and moist heat.[18–21] This study was undertaken to see what effect prolonged, continuous exposure of polyurethane elastomeric ligatures to disinfectant solutions has upon their strength and glass transition temperature. Tensile strength is an important property of elastic ligatures because maintenance of force delivery is needed to sustain full engagement of archwires in the bracket slot for tooth movement.[1] The decrease in tensile load at failure after exposure to disinfectant solution observed in this study parallels some of the results from Jeffries and von Fraunhofer.[3] This is suggestive that the aqueous component or the chemical substances in the disinfectant solution may plasticize or cause degradation of the elastomers.

The glass transition temperature is often used as a measure to investigate the structure and mechanical properties of polymeric materials.[22] Although the actual structure of the orthodontic elastomeric ligatures is proprietary information, the glass transition temperature can provide insights into its molecular configura-

Table 1. Glass Transition Temperatures (in °C) of Elastomeric Ligatures Exposed to Disinfectants

	Manufacturer*					
	American Orthodontics (AO)		3M Unitek (3M)		Rocky Mountain Orthodontics (RMO)	
Exposure Time	Cidexplus	Vital Defense-D	Cidexplus	Vital Defense-D	Cidexplus	Vital Defense-D
Control	−45.8 ± 0.8		−44.4 ± 0.7		−48.0 ± 0.1	
14 Days	−46.4 ± 0.3**	−43.0 ± 0.4	−45.3 ± 0.5	−41.8 ± 0.4	−47.0 ± 1.2	−42.9 ± 0.3
28 Days	−46.6 ± 0.4	−42.3 ± 0.2	−45.7 ± 0.5	−41.3 ± 0.4	−46.3 ± 0.6	−42.5 ± 0.3

* Three-way analysis of variance found significant differences within manufacturer ($P < .0001$), disinfectant ($P < .0001$), and time exposure ($P < .05$).

** With the exception of the AO ligatures exposed to Cidexplus for 14 days, all other exposure groups exhibited significantly ($P < .05$) different glass transition temperatures (T_g) from the respective unexposed ligatures (Dunnett's post hoc test).

tion or if structural changes occurred. This study showed a change in glass transition temperature after prolonged exposure to the solutions, which suggests disruption of the intermolecular bonds. Renick et al[15] studied polyurethane elastomeric chains exposed to the oral environment for orthodontic treatment and showed a reduction in glass transition temperature. In the current study, it is odd to discover that the disinfectant solution used affected the glass transition temperature differently. For the AO and 3M ligatures, the glass transition temperature became more negative with Cidexplus, while it became less negative with Vital Defense-D. The pattern was consistent and continued over longer exposure times from 14 to 28 days. The glass transition temperature of the RMO ligatures, however, became less negative with exposure. With plasticization, one would expect a lowering of the glass transition temperature. An increase in glass transition temperature may indicate a different form of degradation occurred that needs further investigation.

The degradation of the elastic ligatures as seen in the decrease in strength and glass transition temperature may possibly be caused by the aqueous disinfectant solution acting as a plasticizer on the ligature polymer. The mechanism of action by which these disinfectant solutions destroy the bacteria is by acting as a surface active agent (surfactant) or detergent, which is also lethal to the organism. Also, in the process of storing these ligatures in solution, water gets incorporated into the polymer. Both water and detergents have a plasticizing effect on most polymers, which causes the polymeric chains to slip past each other, especially under load. The combination of a surfactant and water would be an especially potent plasticizer. As noted above, a different mechanism may also be responsible for some of the changes. Huget et al[21] exposed orthodontic elastomers to distilled water for 1, 7, 14, 42, and 70 days and examined the storage solutions for leached organic substances. They found leached organic substances only after 14 days of storage, indicating a time-dependent degradation occurs in orthodontic polyurethanes. They speculated that water absorption first leads to plasticization followed by chemical degradation of the polymer backbone with longer exposure times.

Time of exposure was a significant factor in this study. The strength values of the elastomeric ligatures, regardless of manufacturer, were at the highest within the first hour of disinfection and gradually decreased. Still, in most cases, the strength values tended to plateau after eight hours of storage. That most commercially available disinfectants require more than one hour of disinfection to achieve antibacterial effectiveness clinically means that any elastomer exposed to disinfectant solution for sterilization may have its quality compromised. However, the decrease in strength observed between the unexposed and exposed elastomers is only a fraction of the original strength. Beyond eight hours of storage, the strength, on average, was approximately 79%, 86%, and 70% of the original strength for the AO, 3M and RMO ligatures, respectively. These strength degradations may not be clinically significant when one considers the force relaxation normally encountered with usage. A positive aspect, however, may be encountered with the plasticization of the ligatures after exposure to disinfectant. As long as the tensile strength is not surpassed, a less rigid, plasticized ligature should be more easily stretched over the bracket wings, thereby increasing ease in application. A clinical study, however, would be useful to determine if the breakage rate at tie-in of the ligatures is different between cold sterilized and as-received ligatures.

CONCLUSIONS

a. Compared to unexposed specimens, tensile load to failure of elastomeric ligatures decreased when exposed to disinfectant solution for one hour or more.
b. Glass transition temperature of ligatures changed over longer exposure times to disinfectant solution.
c. Ligature tensile load at failure and glass transition temperature followed the pattern: 3M Unitek > American Orthodontics > Rocky Mountain Orthodontics.

REFERENCES

1. Eliades T, Eliades G, Watts DC, Brantley WA. Elastomeric ligatures and chains. In: Brantley WA, Eliades T, eds. *Orthodontic Materials: Scientific and Clinical Aspects.* Stuttgart: Thieme; 2001:174–187.
2. Mayberry D, Allen R, Close J, Kinney DA. Effects of disinfection procedures on elastomeric ligatures. *J Clin Orthod.* 1996;3:49–51.
3. Jeffries CL, von Fraunhofer JA. The effects of 2% alkaline glutaraldehyde solution on the elastic properties of elastomeric chain. *Angle Orthod.* 1991;61:25–30.
4. Andreasen GF, Bishara S. Comparison of alastik chains with elastics involved with intra-arch molar forces. *Angle Orthod.* 1970;40:151–158.
5. Bishara SE, Andreasen GF. A comparison of time-related forces between plastic alastiks and latex elastics. *Angle Orthod.* 1970;40:319–328.
6. Hershey HG, Reynolds WG. The plastic module as an orthodontic tooth-moving mechanism. *Am J Orthod.* 1975;67:554–562.
7. Wong AK. Orthodontic elastic materials. *Angle Orthod.* 1976;46:196–205.
8. Ash JL, Nikolai RJ. Relaxation of orthodontic elastomeric chains and modules in vitro and in vivo. *J Dent Res.* 1978;57:685–690.
9. Stevenson JS, Kusy RP. Force application and decay char-

acteristics of untreated and treated polyurethane elastomeric chains. *Angle Orthod.* 1994;64:455–467.
10. De Genova DC, McInnes-Ledoux P, Weinberg R, Shaye R. Force degradation of orthodontic elastomeric chains—a product comparison study. *Am J Orthod.* 1985;87:377–384.
11. Baty DL, Storie DJ, von Fraunhofer JA. Synthetic elastomeric chains: a literature review. *Am J Orthod Dentofacial Orthop.* 1994;105:536–542.
12. Kohn WG, Collins AS, Cleveland JL, Harte JA, Eklund KJ, Malvitz DM. Guidelines for infection control in dental healthcare settings-2003. *MMWR Morbidity and Mortality Weekly Report.* 2003;52(RR17):1–61.
13. Rosen SL. *Fundamental Principles of Polymeric Materials. 2nd ed.* New York: John Wiley and Sons; 1993:9–51,103–119.
14. Wendlandt WWM. *Thermal Analysis.* 3rd ed. New York: John Wiley and Sons; 1986:424–442,23–59.
15. Renick MR, Brantley WA, Beck FM, Vig KW, Webb CS. Studies of orthodontic elastomeric modules. Part I: Glass transition temperatures for representative pigmented products in the as-received condition and after orthodontic use. *Am J Orthod Dentofacial Orthop.* 2004;126:337–343.
16. Russell KA, Milne AD, Khanna RA, Lee JM. In vitro assessment of the mechanical properties of latex and nonlatex orthodontic elastics. *Am J Orthod Dentofacial Orthop.* 2001;120:36–44.
17. Eliades T, Eliades G, Watts DC. Structural conformation of in vitro and in vivo aged orthodontic elastomeric modules. *Eur J Orthod.* 1999;21:649–658.
18. Phua SK, Castillo E, Anderson JM, Hiltner A. Biodegradation of a polyurethane in vitro. *J Biomed Mater Res.* 1987;21:231–246.
19. Schollenberger CS, Stewart FD. Thermoplastic polyurethane hydrolysis stability. *J Elastoplast.* 1971;3:28–56.
20. Magnus G, Dunleavy RA, Critchfield EF. Stability of urethane elastomers in water, dry air and moist air environments. *Rubber Chem Tech.* 1966;39:1328–1333.
21. Huget EF, Patrick KS, Nunez LJ. Observations on the elastic behavior of a synthetic orthodontic elastomer. *J Dent Res.* 1990;69:496–501.
22. Sperling LH. *Introduction to Physical Polymer Science.* 2nd ed. New York: John and Wiley and Sons; 1992:1–65,303–382.