Effect of Water Storage on the Flexural Properties of Three Glass Fiber–Reinforced Composites

John Chai, BDS, MS, MJ, DLaw[a]/Yutaka Takahashi, DDS, PhD[b]/Kazuhiro Hisama, DDS, PhD[c]/
Hiroshi Shimizu, DDS, PhD[d]

Purpose: The effect of water sorption on the flexural strength and flexural modulus of three fiber-reinforced composites was studied. ***Materials and Methods:*** Bar-shaped specimens of each material were prepared according to the manufacturers' instructions. The flexural strength and modulus of each specimen were tested after the specimens were immersed in distilled water for 1, 7, 60, and 180 days. SEMs were taken to examine the mode of failure. The volume percentage of fiber content of each fiber-reinforced composite was experimentally estimated. ***Results:*** The ascending order of flexural strength and modulus among the materials was generally: FibreKor < Stick < Vectris. The flexural strengths of Stick and FibreKor specimens at 1 and 180 days were not significantly different. Although the 180-day Vectris specimens possessed significantly lower flexural strength than the 1-day specimens, the flexural strengths of Vectris specimens at 1, 7, 60, and 180 days remained significantly higher than those of Stick and FibreKor. The difference in flexural modulus for each fiber-reinforced composite related to the duration of water immersion was not significant. ***Conclusion:*** Water immersion affected the flexural strengths of the three composites to a different degree but did not affect their flexural moduli significantly. For each duration of water immersion, the flexural property of the fiber-reinforced composite, in ascending order of significant difference, was: FibreKor < Stick < Vectris. *Int J Prosthodont 2005;18:28–33.*

Several factors act in concert to determine the strength of fiber-reinforced composites (FRC). Incidentally, many such factors can be affected by the aqueous oral environment. The FRC derives its strength from the innate superior mechanical properties of the glass fibers. Increasing the fiber-to-matrix volume ratio favors the strength of the FRC.[1,2] However, impregnation of the fibers with the matrix becomes increasingly difficult with a higher volume of fiber.

[a]Professor Emeritus, Northwestern University, Evanston, Illinois.
[b]Professor, Division of Removable Prosthodontics, Fukuoka Dental College, Japan.
[c]Assistant Professor, Division of General Dentistry, Fukuoka Dental College, Japan.
[d]Assistant Professor, Division of Removable Prosthodontics, Fukuoka Dental College, Japan.

Correspondence to: *Dr John Chai, 1065 King's Road, Room 401, Eastern Centre, Quarry Bay, Hong Kong SAR, China. e-mail: jchai@northwestern.edu.*

Glass fibers vary in their surface chemical composition and susceptibility to hydrolytic degradation. Glass fibers containing alkali and earth alkali oxides are known to degrade mechanically by hydrolytic action.[3] Boron oxide, a glass-forming agent present at 6 wt% to 9 wt% in E-glass fibers and < 1 wt% in S-glass fibers, is also implicated in the hydrolytic degradation of the glass fiber.[3,4] E- and S-glass fibers are most commonly found in FRCs for dental use. It is hypothesized that B_2O_3 leaches from the glass-fiber surface and affects its strength by disrupting the glass-supporting network.[3]

It is well-established that water storage decreases the strength of resin polymers.[5–7] Water molecules diffuse inward into the FRC resin matrix and act as plasticizers to facilitate the movement of resin polymer chains under stress, thus adversely affecting the strength of the polymer. The amount of water sorption into the FRC matrix is affected by several factors. Water sorption generally increases with a decrease in the percentage fiber content of the FRC. This is due to an

Table 1 Fiber-Reinforced Composite (FRC) Materials Studied

FRC core material	Lot No.	Composition	Fabrication procedure
Vectris Pontic, Ivoclar Vivadent	D94030	bis-GMA (24.5%), triethyleneglycol dimethacrylate (6.2%), decandiol dimethacrylate (0.3%), urethane dimethacrylate (0.1%), highly dispersed silica (3.5%), catalysts and stabilizers (< 0.3%), pigments (< 0.1%), preimpregnated E-glass fibers (65.0%)	Initial polymerization for 1 min with light-curing unit (Targis Quick, Ivoclar Vivadent); final polymerization in light- and heat-curing unit (Targis Power, Ivoclar Vivadent) for 25 min
FibreKor Jeneric/Pentron	57486	Preimpregnated S-glass fibers (≈ 60%) in 100% bis-GMA matrix	Initial polymerization for 1 min with light-curing unit (Alfa Light II, Morita); final polymerization in light-curing unit (Alfa Light II) for 15 min
Stick, StickTech	1010321-R-0058	E-glass fibers impregnated with PMMA	Wetting of fibers with Stick Resin (StickTech); polymerization as for FibreKor

increase in the relative portion of water-absorbing polymer matrix in the FRC as the percentage fiber content decreases.[8] The hydrophilicity of the matrix polymer also affects water sorption. More hydrophilic resins, such as 2-hydroxyethyl methacrylate (HEMA) and bisphenol glycidyl methacrylate (bis-GMA), absorb more water than does polymethyl methacrylate (PMMA).[9] Furthermore, insufficient impregnation of fibers by the matrix leaves air voids in the FRC, which weakens the FRC by directly affecting its mechanical strength and indirectly increasing its susceptibility to water sorption.[3,8,9]

A few commercially available FRC systems, such as Vectris, FibreKor, and Stick, are based on the use of impregnated or preimpregnated glass fibers and standardized systems of fabrication to maximize fiber compaction and minimize air voids. The purpose of this study was to investigate the effect of water immersion on the flexural strength and modulus of representative products of these three systems. The hypothesis was that there would be no significant differences in these properties among the systems.

Materials and Methods

Three FRCs—Vectris Pontic, FibreKor, and Stick—were selected for the study (Table 1). The experimental protocol was adapted from ISO 10477:1992(E) for polymer-based crown and fixed partial denture materials.[10] Thirty-two bar-shaped specimens of each material were polymerized according to the manufacturers' instructions in a Teflon (DuPont) mold with a cavity of 2 mm × 2 mm × 25 mm. The materials were polymerized initially in the mold under a slide glass cover and then removed from the mold for final polymerization (Table 1). The accuracy of the dimensions was verified with a micrometer at three locations of each dimension to within 0.01-mm tolerance. The specimens were divided into four groups of eight for each material and immersed in distilled water at 37 ± 1°C for 1, 7, 60, and 180 days, respectively.

The flexural strength and modulus of each specimen were tested after immersion. Each specimen was placed on a support with a 20-mm span for the three-point flexural test. A vertical load was applied at the midpoint of each specimen at a cross-head speed of 10 mm/min on a load-testing machine (TCM-200, Minebea). The load and deflection of the specimen were recorded on a chart recorder. The flexural strength of each specimen, in MPa, was calculated with the following formula[10]:

$$3PL/2bd^2$$

where P = maximum load; L = span distance (20 mm); b = width of the specimen (2 mm); and d = thickness of the specimen (2 mm). The load P was manually read from each load/deflection graph.

Flexural modulus (E), in GPa, was calculated from the following formula:

$$FL^3/4bd^3D$$

where F = load (N) at a convenient point in the straight-line portion of the load/deflection graph; and D = deflection (mm) at load F.

The data were analyzed statistically using two-way analysis of variance (ANOVA) to detect the effects caused by material and immersion time (Statistica, StatSoft). One-way ANOVA and Newman-Keuls post hoc comparison were applied when appropriate (95% confidence level). All tests were performed under uniform atmospheric conditions of 23.0 ± 1°C and 50% ± 1% relative humidity.

Fractured gross specimens and scanning electron micrographs (SEM) of the fractured surfaces of representative specimens were examined for the mode of fracture.

The fiber content (vol%) of each FRC was determined with an ashing method.[11] Four specimens of each FRC were desiccated for 36 hours at 37°C and weighed to

Table 2 Flexural Strength (MPa) of Fiber-Reinforced Composites Tested*

Material	1 d		7 d		60 d		180 d	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Vectris	696[a]	22	671[a,b]	21	655[b]	35	604	35
FibreKor	367[f,g]	29	405[d,e]	28	381[e,f]	29	343[g]	23
Stick	454[c]	19	460[c]	19	447[c]	24	430[c,d]	22

*Groups with the same superscripted letter showed no statistically significant difference (one-way ANOVA, Neuman-Keuls; $P > .05$).
SD = standard deviation.

Table 3 Flexural Modulus (GPa) of Fiber-Reinforced Composites Tested

Material	1 d		7 d		60 d		180 d		All periods	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Vectris	30.2	1.6	28.9	30.0	30.0	3.9	30.0	2.2	29.6	2.5
FibreKor	22.3	1.5	25.6	22.5	22.5	2.4	22.5	1.3	23.8	2.2
Stick	27.2	2.4	27.8	28.3	28.3	3.6	28.3	2.6	28.0	2.6

SD = standard deviation.

an accuracy of 1 mg. The specimens were then ashed for 45 minutes at 700°C. The weight of each specimen was measured with an electronic scale before and after ashing. The fiber content (V_g, vol%) was calculated with the following formula:

$$W_g/\rho_g \div (W_g/\rho_g + W_r/\rho_r)$$

where W_g = wt% of fiber; ρ_g = density of fiber (Vectris 2.53 g/cm^3, FibreKor 2.43 g/cm^3, Stick 2.54 g/cm^3); W_r = wt% of matrix; and ρ_r = density of matrix (Vectris 1.18 g/cm^3, FibreKor 1.18 g/cm^3, Stick 1.19 g/cm^3).[9,12] One-way ANOVA and Newman-Keuls post hoc comparison were applied to the data (95% confidence level).

Results

Two-way ANOVA revealed significant differences ($P <$ .05) in flexural strength attributed to the material type, duration of immersion, and interaction between these two variables. The flexural strength among the materials for each immersion period was statistically significant (Table 2; one-way ANOVA and Newman-Keuls). For each duration of water immersion, flexural strength of the FRCs, in ascending order of significant difference, was: FibreKor < Stick < Vectris. The flexural strength of Vectris decreased gradually with increasing immersion time. Its flexural strength at 180 days (604 MPa) was significantly lower than at 1 day (696 MPa). The flexural strength of FibreKor and Stick fluctuated with water immersion, but the differences at 1 and 180 days were not significantly different.

Two-way ANOVA revealed significant differences (P < .05) in flexural modulus (E) attributed to the material type only (Table 3). E of all three materials combining all immersion periods was significantly different in the following ascending order of E: FibreKor (23.8 ± 2.2 GPa) < Stick (28.0 ± 2.6 GPa) < Vectris (29.6 ± 2.5 GPa) (Table 3; one-way ANOVA and Newman-Keuls).

Visual inspection of the fractured specimens indicated that all specimens remained in one piece, and the appearance of the fractures was similar. Breakage of fibers was not observed on the undersurface (tensile surface) of any specimen. The zone of fiber fracture and detachment[13] on representative specimens was limited to the area around the application of the compressive load (Fig 1). The fracture line manifested as breakages of fibers that traversed the width of the specimens on the loading surface, which was interrupted at times by separation between the fibers that traversed the length of the specimens (Fig 1).

SEMs of the three types of FRC specimens revealed similar modes of failure (Figs 2 to 4). Fiber fracture was the predominant mode of failure of all FRC specimens. Fibers remained adhered to the matrix after fracture, indicating good fiber-matrix bonding. Matrix fracture appeared to be localized where the fibers fractured.

The fiber contents (vol%) of Vectris, FibreKor, and Stick were, respectively, 46.5% ± 0.6%, 45.2% ± 1.3%, and 51.8% ± 1.3%. Stick possessed a significantly higher fiber content than either Vectris or FibreKor. The fiber content of Vectris and FibreKor was not significantly different.

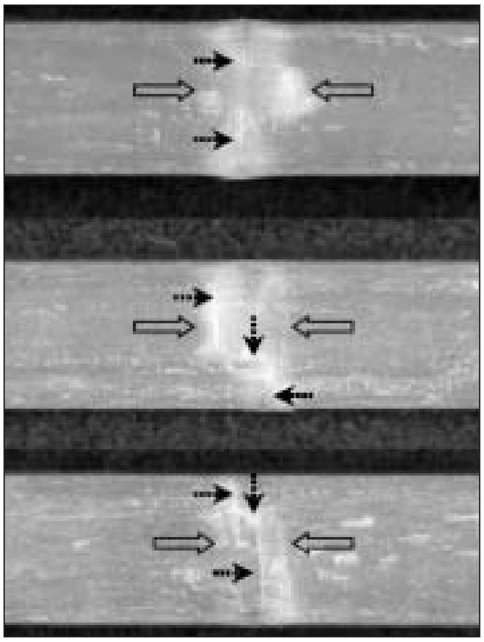

Fig 1 Representative specimens of Vectris *(top),* FibreKor *(center),* and Stick *(bottom); open arrows* = zone of fiber fracture and detachment; *dashed arrows* = fracture line.

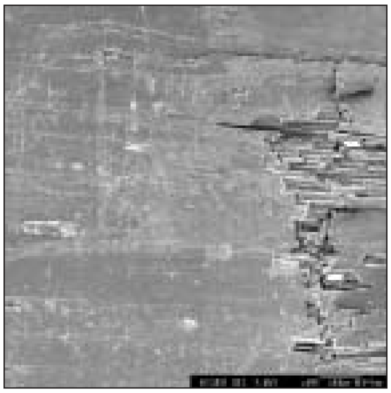

Fig 2 SEM view of Vectris depicts fiber fracture *(arrowheads);* fibers are held together by matrix after their fracture *(arrows).*

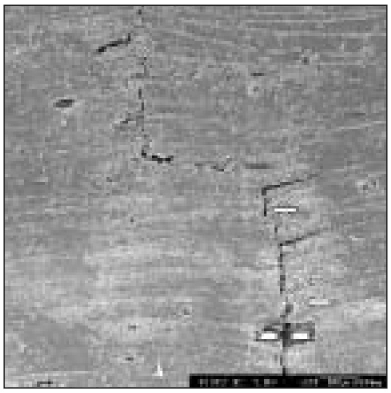

Fig 3 SEM view of FibreKor depicts fiber fracture *(arrowheads);* fibers are held together by matrix after their fracture *(arrows).*

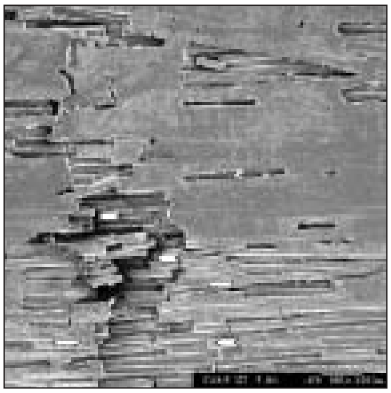

Fig 4 SEM view of Stick depicts fiber fracture *(arrowheads);* fibers are held together by matrix after their fracture *(arrows).*

Discussion

Compared to 1 day, 180 days of water immersion decreased the flexural strength of Vectris significantly; its value (604 MPa) remained significantly higher than those of FibreKor (343 MPa) and Stick (430 MPa), which were not significantly different from their 1-day values. The flexural strength of Vectris and FibreKor unidirectional FRCs has been shown to decrease significantly after water immersion for 30 days.[9] Tandem experiments also showed that flexural strength of E-glass fiber–reinforced denture acrylic resins decreases significantly with increasing immersion time[3,11] but tends to stabilize after 4 weeks.[11] Although 180 days of immersion is a reasonable length of time, it is unknown whether Vectris, with a decreasing trend in flexural strength, or FibreKor, with an apparently fluctuating flexural strength, has stabilized. Conversely, the flexural strength of Stick appeared to remain stable throughout the entire immersion period.

Immersion time did not significantly affect the flexural modulus of the FRCs. This is congruent with earlier findings that water storage generally has a minimal effect on the modulus of fiber-reinforced resins.[3,11]

Vectris generally displayed significantly higher flexural strength than Stick, the strength of which was, in turn, significantly higher than that of FibreKor. A discussion to account for the difference should include the variation of the FRCs in water sorption, and the type and percentage of fiber content.

A recent study revealed an ascending order of increasing water sorption among three unidirectional FRCs to be: Vectris < FibreKor < Stick.[14] SEM observation suggested that the heat- and light-polymerization process for Vectris might have helped to decrease flaws and porosities included during specimen preparation. Hence, water sorption into such inclusion defects would have been minimized. Furthermore, the preimpregnation of Vectris and FibreKor fibers likely also minimizes voids at the interface.[14] As mentioned earlier, voids and defects weaken the strength of the FRC by directly affecting the material structurally and indirectly promoting water sorption. Water sorption by capillary action through cracks and voids along improperly bonded fiber-matrix interfaces is estimated to be 450 times faster than water diffusion through the resin matrix.[15] Thus, the proper impregnation and bonding of fibers not only ensures effective load transfer from the matrix to the fiber,[9] it prevents accelerated water sorption through capillary action at the fiber-matrix interface.

Vectris's superior flexural strength could be explained by the lowest water sorption among the three FRCs and the minimal amount of flaws and voids within its matrix and at the fiber-matrix interface. This was evident even when viewed in the context of Vectris's significantly lower fiber content (46.5%) in comparison with Stick (51.8%). The quality of the Vectris fiber-matrix interface might have also shielded its hydrolytically susceptible E-glass fibers from any significant degradation. Despite its higher fiber content, Stick's flexural strength was significantly lower than that of Vectris.

Although still impressive, the flexural strength displayed by FibreKor, in the upper 300 MPa range, was the lowest among the three FRCs studied. FibreKor was intermediate in water sorption compared to Vectris and Stick.[14] Water immersion appears to be more detrimental to an S-glass fiber acrylic resin composite than to an E-glass fiber composite. That study also suggested that the fiber-matrix adhesion of the S-glass fiber composite is more severely weakened with water immersion.[3] The extent to which such findings can be applied to the present study is unknown, as the materials are not identical. Furthermore, cross-sectional SEMs of the present materials did not reveal any distinctive signs of compromised adhesion between the FibreKor S-glass fibers and its matrix.[14] Thus, a plausible explanation for FibreKor's lowest strength among the three FRCs could be its moderate level of water sorption and that its volume percentage of fiber content was the lowest among the three FRCs.

This study indicated that the flexural strengths of the three FRCs after a moderately long duration of water immersion, 180 days, were approximately between 350 and 600 MPa. This range of strength compares favorably with the flexural strengths of a high-purity alumina (Procera, Nobel Biocare), glass-infiltrated alumina (In-Ceram, Vita), and zirconium-reinforced glass-infiltrated alumina (In-Ceram Zirconia, Vita) all-ceramic core material.[16–18] Short- and intermediate-term clinical studies on these FRCs have not revealed significant failure attributed to the strength of the core material, although complications such as wear, discoloration, and delamination of the veneer composite existed.[19–24] Thus, it appears that the strength of these FRC core materials is promising, whereas other aspects of these systems still require improvement.

Conclusion

Water immersion did not significantly affect the flexural modulus of the three FRCs. For each duration of water immersion, the flexural property of the FRCs, in ascending order of significant difference, was: FibreKor < Stick < Vectris. The 180-day Vectris specimens possessed significantly lower flexural strength than the 1-day specimens. The flexural strengths of Stick and FibreKor specimens at 1 and 180 days were not significantly different. The differences were explained by the variation of the FRCs in water sorption, and the type and percentage of fiber content.

References

1. Karmaker AC, DiBenedetto AT, Goldberg AJ. Continuous fiber reinforced composites as alternatives for metal alloys used for dental appliances. J Biomater Appl 1997;11:318–328.
2. Vallittu PK, Lassila VP, Lappalainen R. Acrylic resin-fiber composite—Part I: The effect of fiber concentration on fracture resistance. J Prosthet Dent 1994;71:607–612.
3. Vallittu PK, Ruyter IE, Ekstrand K. Effect of water storage on the flexural properties of E-glass and silica fiber acrylic resin composite. Int J Prosthodont 1998;11:340–350.
4. Vallittu PK. Compositional and weave pattern analyses of glass fibers in dental polymer fiber composites. J Prosthodont 1998;7:170–176.
5. Takahashi Y, Chai J, Kawaguchi M. Effect of water sorption on the resistance to plastic deformation of a denture base material relined with four different denture reline materials. Int J Prosthodont 1998;11:49–54.
6. Chai J, Takahashi Y, Kawaguchi M. The flexural strengths of denture base acrylic resins after relining with a visible-light activated material. Int J Prosthodont 1998;11:121–124.
7. Takahashi Y, Chai J, Kawaguchi M. Equilibrium strengths of denture polymers subjected to long-term water immersion. Int J Prosthodont 1999;12:348–352.
8. Miettinen VM, Narva KK, Vallittu PK. Water sorption, solubility and effect of post-curing of glass fibre reinforced polymers. Biomaterials 1999;20:1187–1194.
9. Behr M, Rosentritt M, Lang R, Handel G. Flexural properties of fiber reinforced composite using a vacuum/pressure or a manual adaptation manufacturing process. J Dent 2000;28:509–514.
10. International Organisation for Standardization. ISO 10477 for Dentistry 1992(E). Polymer-Based Crown and Bridge Materials. Geneva: International Organization for the Testing of Materials, 1992.

11. Vallittu PK. Flexural properties of acrylic resin polymers reinforced with unidirectional and woven glass fibers. J Prosthet Dent 1999;81:318–326.
12. Vallittu PK. Effect of 180-week water storage on the flexural properties of E-glass and silica fiber acrylic resin composite. Int J Prosthodont 2000;13:334–339.
13. Chong KH, Chai J. Strength and mode of failure of unidirectional and bidirectional glass fiber–reinforced composite materials. Int J Prosthodont 2003;16:161–166.
14. Chai J, Takahashi Y, Hisama K, Shimizu H. Water sorption and dimensional stability of three glass fiber–reinforced composites. Int J Prosthodont 2004;17:195–199.
15. Jancar J, DiBenedetto AT, Goldberg AJ. Thermoplastic fibre-reinforced composites for dentistry. Part II. Effect of moisture on flexural properties of unidirectional composites. J Mater Sci Mater Med 1993;4:562–568.
16. Wen MY, Mueller HJ, Chai J, Wozniak WT. Comparative mechanical property characterization of three all-ceramic core materials. Int J Prosthodont 1999;12:534–541.
17. Esquivel-Upshaw JF, Chai J, Sansano S, Shonberg D. Resistance to staining, flexural strength, and chemical solubility of core porcelains for all-ceramic crowns. Int J Prosthodont 2001;14:284–288.
18. Chong K, Chai J, Takahashi Y, Wozniak WT. Flexural strength of In-Ceram Alumina and In-Ceram Zirconia core materials. Int J Prosthodont 2002;15:183–188.
19. Göhring T, Mörmann WH, Lutz F. Clinical and scanning electron miscroscopic evaluation of fiber-reinforced inlay fixed partial dentures: Preliminary results after one year. J Prosthet Dent 1999;82:662–668.
20. Vallittu PK, Sevelius C. Rein-bonded, glass fiber-reinforced composite fixed partial dentures: A clinical study. J Prosthet Dent 2000;84:413–418.
21. Freilich MA, Meiers JC, Duncan JP, Eckrote KA, Goldberg AJ. Clinical evaluation of fiber-reforced fixed bridges. J Am Dent Assoc 2002;133:1524–1534.
22. Göhring TN, Schmidlin PR, Lutz F. Two-year clinical and SEM evaluation of glass-fiber-reinforced inlay fixed partial dentures. Am J Dent 2002;15:35–40.
23. Bohlsen F, Kern M. Clinical outcome of glass-fiber-reforced crowns and fixed partial dentures: A three-year retrospective study. Quintessence Int 2003;34:493–496.
24. Behr M, Rosentritt M, Handel G. Fiber-reinforced composite crowns and FPDs: A clinical report. Int J Prosthodont 2003;16:239–243.

Literature Abstract

Postprocedural problems in an overdenture population: A longitudinal study

Overdenture therapy has become an accepted and realistic alternative to extraction of all remaining teeth. This longitudinal study evaluated the difference between people who experienced postprocedural problems in abutment teeth with overdenture therapy to those who had no problems. All patients treated in the prosthodontics clinic at the University of Iowa College of Dentistry from 1973 to 1994 were included in this prospective clinical longitudinal cohort study. At recall appointments, the overdenture abutment teeth were evaluated by a single examiner for the condition of the restorations, dental caries, and periodontal conditions. The result showed 81 abutment failures in 51 subjects, or a failure rate of 121 per 1,000 abutment teeth (12.1%) or 186 per 1,000 subjects at risk (18.6%). The most common cause of failure (37.0%) was endodontically treated teeth developing periradicular lesions. This was usually a result of recurrent caries causing the loss of restorations sealing the root canal of the endodontically treated teeth, thus disrupting the occlusal seal. Result also showed 25 vertical root fractures in 20 subjects. Sixteen of the fractures were in maxilla, 21 were in males, and 13 were opposed by natural teeth. Chi-square analysis found only that overdentures in the maxillary arch (P = .04) and those opposed by natural dentition (P = .05) were a significant risk for vertical fracture. The highest risk of failure was caused by inadequate oral hygiene and failure to use fluoride consistently, which resulted in caries, loss of the restoration and, consequently, a periradicular lesion. It was concluded that improved communication between the patient and the dentist with regard to oral hygiene practices, as well as regular recall appointments, are critical to the success of overdenture therapy.

Ettinger RL, Qian F. *J Endodon* 2004;30:310-–314. **Reference:** 16. **Reprint:** Dr. Ettinger, Professor, Department of Prosthodontics and Dows Institute for Dental Research, University of Iowa, Iowa City, IA 52242. e-mail: ronald- ettinger@uiowa.edu—*Myung W. Brian Chang, Lincoln, NE*