Dental Materials

self CDE 2 HOURS CREDIT instruction

Comparative study of the fluorescence intensity of dental composites and human teeth submitted to artificial aging

Tatiana Jablonski, DDS ▪ Marcos Kenzo Takahashi, DDS, MSD ▪ Rafael Torres Brum, DDS, MSD
Rodrigo Nunes Rached, DDS, MSD, PhD ▪ Evelise M. Souza, DDS, MSD, PhD

The aim of this study was to evaluate quantitatively the fluorescence of resin composites and human teeth, and to determine the stability of fluorescence after aging. Ten specimens were built using a 1 mm thick increment of dentin composite overlapped by a 0.5 mm thick increment of enamel composite. Ten sound human molars were sectioned and silicon carbide-polished to obtain enamel and dentin slabs 1.5 mm in thickness. Fluorescence measurements were carried out by a fluorescence spectrophotometer before and after thermocycling (2000 cycles, 5°C and 55°C). One-way analysis of variance with repeated measures and Tukey's test were performed at a significance level of 5%. Most of the tested composites showed significant differences in fluorescence both before and after aging ($P < 0.05$). Opallis was the only composite whose fluorescence was similar to that of human teeth at both periods of evaluation ($P > 0.05$), and was the only composite that showed comparable results of fluorescence to the tooth structure before and after thermocycling. With the exception of Filtek Supreme, there were significant reductions in fluorescence intensity for all the tested composites ($P < 0.05$).

Received: May 21, 2012
Accepted: December 6, 2012

Fluorescence is one of the most important optical properties of dental materials and is defined as an immediate physical phenomenon, occurring in less than 10^{-8} sec, in which the material absorbs ultraviolet (UV) rays invisible to the human eye and emits visible light, mainly in the blue spectrum.[1]

Reproducing the natural layering of dental tissues is a challenge to the dental clinician because different light sources emit UV rays at different intensities; this results in different patterns of fluorescent reflection of teeth and restorative materials.[2-4] Natural teeth appear whiter and brighter in daylight because of the large amount of light in the blue spectrum reflected as a result of fluorescence.[5-7] However, there is a significant UV component not only in sunlight but also in certain fluorescent bulbs, photo flash bulbs, high intensity studio lights, and entertainment lights known as black lights.[8] The latter type of light emits filtered 350 nm and 400 nm UV rays that are reflected as visible light and result in an emission spectrum in the intense blue region when absorbed by a fluorescent body.[9]

In dental porcelains, fluorescence is achieved by incorporating small amounts of inorganic oxides of rare earths with fluorescent properties.[10,11] These luminophores are usually composed of cerium, europium, terbium, ytterbium, dysprosium, and samarium.[8,12] None of these oxides alone can impart fluorescence similar to that of human teeth to porcelains but must instead be blended in specific concentrations.[6,7] It is known that at least 1 of these elements (ytterbium) is being used as a luminophore in 1 of the evaluated commercial composite resins (4 Seasons, Ivoclar Vivadent, Inc.), according to the manufacturer. However, most manufacturers don't reveal which compounds are responsible for the fluorescence of their products.

Fluorescence spectrophotometry has been widely used to determine the fluorescence of dental porcelains.[6,8,11,12] Some studies have used the same method to determine differences between the fluorescence of composites and teeth.[13-15]

Since resin composites are constantly affected by physical and chemical agents acting in the oral cavity, changes in their optical properties are easily detected. Previous studies have investigated the color stability of resin composites after accelerated aging, an artificial method in which the material is submitted to extreme conditions using UV light, changes in temperature, and water storage.[16-22] However, this type of aging results in a drastic reduction of the fluorescence intensity of composites.[15,20] Thermocycling is a method that reproduces in vitro oral conditions with successive immersions in water at 5°C and 55°C to induce aging of the material. Previous studies have investigated the effect of thermocycling on the color stability, translucency, and contrast parameters of composite resins.[23,24] However, to date, the effect of thermocycling on the fluorescence intensity of dental composites has not been investigated.

The objective of this study was to evaluate quantitatively the fluorescence intensity of resin composites compared to human teeth, and determine the effect of artificial aging on the fluorescence of these materials. The 2 null hypotheses tested were that the fluorescence properties of composites are similar to those of human teeth and that thermocycling does not affect the fluorescence intensity of composites.

Materials and methods

Composite specimen preparation

Five microhybrid dental composites, each in 2 different shades, were investigated in this study and are listed with their manufacturers in Table 1. Two polytetrafluoroethylene molds were used to build square-shaped specimens with composite dentin shades (1 x 7 x 7 mm) and with composite enamel shades (0.5 x 7 x 7 mm). The 1 mm thick mold was positioned on a glass slide and a mylar strip, then dentin shade composite was inserted into the mold. Another mylar strip and a glass slide were positioned over the composite in order to flatten the surface. Light curing was carried out with

Table 1. Composites used in the study.

Composite	Manufacturer	Shade	Batch
4 Seasons	Ivoclar Vivadent, Inc.	A2 Dentin	H24150
		A2 Enamel	H19814
Charisma	Heraeus Kulzer	OA2	10200
		A2	10205
Esthet.X	DENTSPLY Caulk	A2-O	503032
		A2	50126
Opallis	FGM Produtos Odontologicos	DA2	310106
		EA2	80206
Vit-l-escence	Ultradent Products, Inc.	A2	B1TM7
		Pearl Amber	B1TLY

Table 2. Mean (SD) and variation (Δ) of fluorescence intensity before and after accelerated aging.

Materials	Before	After	Δ(%)
4 Seasons	489.25 (36.07)^{Aa}	408.61 (72.88)^{Ab}	-16.48
Esthet.X	482.36 (11.72)^{Aa}	409.53 (16.22)^{Ab}	-15.09
Vit-l-escence	376.08 (9.21)^{Ba}	320.59 (9.21)^{Bb}	-14.75
Charisma	299.50 (24.31)^{Ca}	162.24 (10.97)^{Cb}	-45.82
Filtek	68.93 (4.06)^{Da}	74.39 (2.74)^{Da}	7.92
Opallis	254.43 (23.27)^{Ea}	204.91 (4.90)^{Eb}	-19.46
Teeth	232.99 (36.22)^{Ea}	224.36 (52.50)^{Ea}	-3.70

Groups connected by the same lowercase letter in the same row and by the uppercase letter in the same column are not statistically significant ($P > 0.05$).

Chart 1. Confidence intervals of fluorescence intensity of dental composites and tooth structure before and after aging.

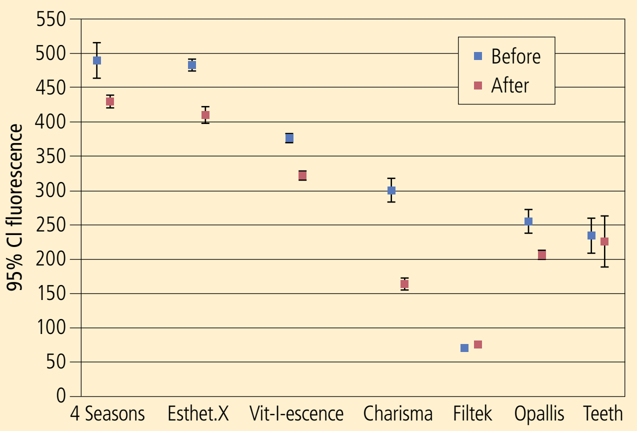
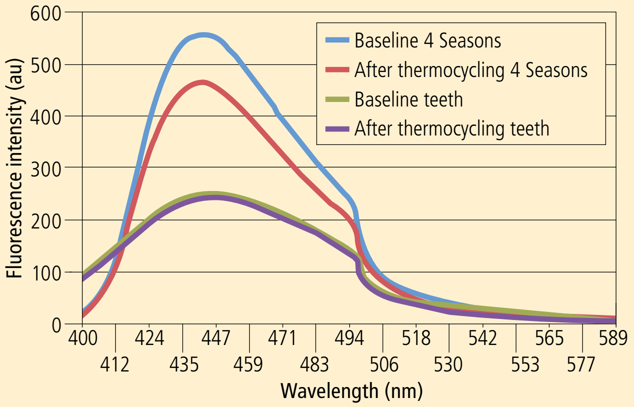


Chart 2. Fluorescence intensity spectra of 4 Seasons and teeth, before (baseline) and after aging.



Abbreviations: au, absorbance units; nm, nanometers.

a quartz-tungsten-halogen curing unit (Optilux 501, Kerr Corporation) at 540 mW/cm^2 for 20 seconds. The top slide and mylar strip were removed, and the 0.5 mm thick mold was positioned for the insertion of the enamel shade composite. A new mylar strip and glass slide were placed on the surface prior to light curing for 20 seconds. The specimens were then removed from the mold, kept in a lightproof receptacle, and stored at 100% humidity (37°C) for 24 hours.

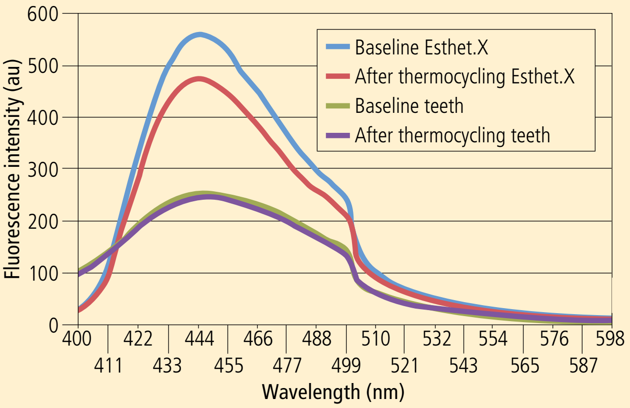
Human teeth specimens

Ten sound human molars were used for enamel/dentin specimens (1.5 x 7 x 7 mm). The buccal surface was cut longitudinally with a diamond saw in a precision cutting machine (Labcut 1010, Extec Corp.) into 2 mm thick slices that were ground and polished to a thickness of 1.5 mm. The thickness of the specimens was verified with a micrometer (103-125, Mitutoyo America Corporation). The final specimens were composed of approximately 0.5 mm enamel and 1 mm dentin. The storage conditions were the same as those described for composite specimens.

Fluorescence measurements

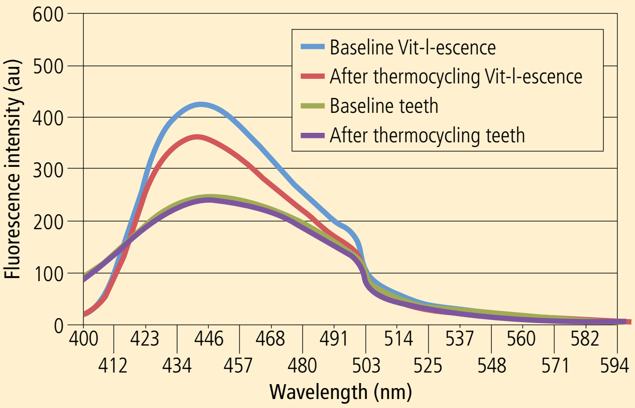
Baseline fluorescence measurements were carried out using a fluorescence spectrophotometer (F-4500, Hitachi High-Technologies Science America, Inc.). The specimens were fixed in an acrylic cuvette and placed inside the spectrophotometer chamber with emission and excitation aperture slits of 2.5 nm. The location of the cuvette allowed the excitation beam to reach the center of the specimen at a wavelength of 380 nm. The values obtained were then used to produce graphs with the aid of the computer software used with the equipment. The fluorescence intensity values used in the present study were within the visible light spectrum and ranged from 400 nm to 600 nm.

Chart 3. Fluorescence intensity spectra of Esthet.X and teeth, before (baseline) and after aging.



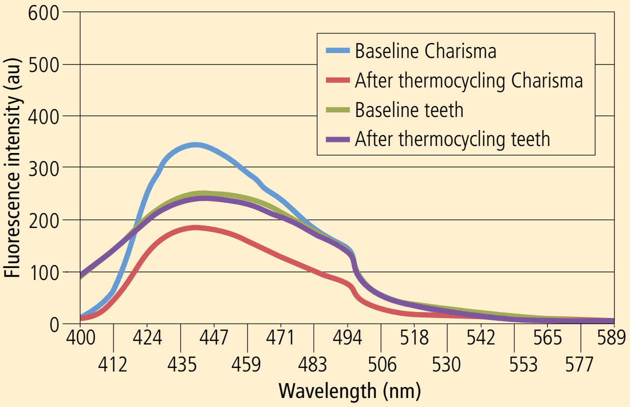
Abbreviations: au, absorbance units; nm, nanometers.

Chart 4. Fluorescence intensity spectra of Vit-l-escence and teeth, before (baseline) and after aging.



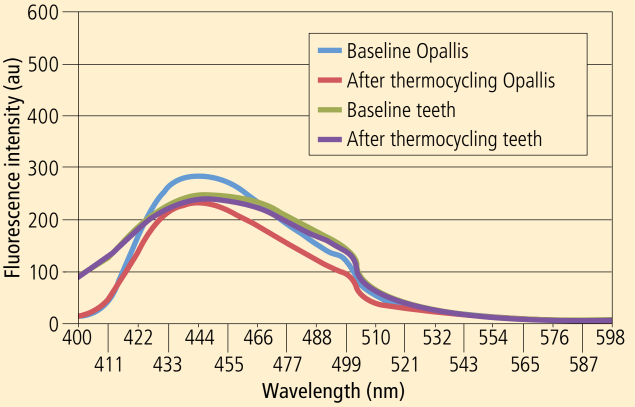
Abbreviations: au, absorbance units; nm, nanometers.

Chart 5. Fluorescence intensity spectra of Charisma and teeth, before (baseline) and after aging.



Abbreviations: au, absorbance units; nm, nanometers.

Chart 6. Fluorescence intensity spectra of Opallis and teeth, before (baseline) and after aging.



Abbreviations: au, absorbance units; nm, nanometers.

Accelerated aging process

After baseline measurements, the specimens were submitted to a thermocycling process with 2000 cycles at temperatures of 5°C and 55°C, with a dwell time of 30 seconds. The fluorescence intensity was measured again using the same method described above.

Statistical analysis

The means of the fluorescence intensity values located in the visible light spectrum between 420 nm and 470 nm were calculated. The data obtained were recorded and submitted to statistical analysis with SPSS 17.0 software (SPSS, Inc.). One-way analysis of variance (ANOVA) with repeated measures and Tukey's test for multiple comparisons were performed at a significance level of 0.05.

Results

The mean fluorescence intensities of resin composites and dental specimens at baseline and after thermocycling are listed in Table 2 with the percentage of variation. Chart 1 displays the 95% confidence interval of fluorescence intensity of the evaluated composites before and after thermocycling. Charts 2-6 illustrate the differences in fluorescence intensity of the composites before and after thermocycling compared to that of tooth structure.

There were significant differences ($P < 0.05$) among groups at baseline and after thermocycling. At baseline, comparisons among different materials demonstrated that composites Esthet.X and 4 Seasons gave the highest fluorescence intensity values, without significant differences from each other ($P > 0.05$). Vit-l-escence showed intermediate fluorescence intensity significantly higher than Opallis and human teeth ($P < 0.05$). Charisma also demonstrated higher fluorescence intensity than human teeth and Opallis, but significantly lower than Vit-l-escence ($P < 0.05$).

After thermocycling, Esthet.X and 4 Seasons had the highest maintained fluorescence intensities, significantly different from the other composites ($P < 0.05$). Charisma had the highest reduction in fluorescence after thermocycling (45%) among all the composites evaluated. With the exception of Filtek Supreme, there were significant reductions in fluorescence intensity for all the tested composites ($P < 0.05$). Enamel/dentin specimens did not show significant differences in fluorescence after aging ($P > 0.05$).

The nanofilled composite Filtek Supreme exhibited significantly lower fluorescence than human teeth before and after aging ($P > 0.05$). Opallis was the only composite that exhibited fluorescence intensity statistically similar to that of tooth structure at both times of evaluation ($P > 0.05$).

Discussion

Although some studies investigated possible ways of incorporating luminophores in composites to increase their fluorescence intensity, the fluorescent components of current resin composites are still mainly unknown.[14,25,26] These components could be incorporated into the organic matrix as well as into the inorganic particles of dental composites. Uo et al demonstrated that the fusion of rare earth oxides to glass filler particles increased the fluorescent properties of an experimental composite.[14] Fluorescent emission by an experimental composite was investigated by including a fluorescent whitening agent composed of an aromatic complex in the polymer matrix.[25] In another study, terbium coordination polymers composed of polyethyl methacrylate resulted in a hybrid polymer structure with high fluorescence.[26]

The manufacturers of only 2 of the evaluated composite resins in this study provided information on their fluorescent components. Ytterbium trifluoride and an organic molecule (not specified by the manufacturer) are claimed to be the luminophores used in 4 Seasons and Opallis composites, respectively. The results of the present study demonstrated that Opallis exhibited fluorescence intensity similar to that of teeth. Esthet.X, 4 Seasons, and Vit-l-escence showed increased fluorescence when compared to human teeth both before and after aging, whereas Charisma showed higher fluorescence than tooth structure only before aging. Filtek Supreme presented the lowest levels of fluorescence in both periods of observation. Because of these discrepancies, the esthetic appearance of dental composite restorations could be compromised due to excess or lack of fluorescence, particularly when such restorations are submitted to irradiation from a light source with a high UV component. After the aging process, most of the evaluated materials showed a significant reduction in fluorescence intensity, except for Filtek Supreme and human teeth. Among all the composites evaluated, Charisma showed the highest reduction of fluorescence. These findings are in accordance with other studies that investigated changes in the opalescence and fluorescence of several composites after accelerated aging carried out with UV light and water spray.[15,20] Lee et al reported reduced fluorescence intensity values and even the absence of fluorescence for some resin-based materials after accelerated aging.[20]

Water sorption, solubility, and diffusion coefficients of resin-based materials have been reported as dependent on the composition of monomers, degree of polymerization of the organic matrix, size and distribution of the filler particles, and properties of the matrix/filler particles interface.[27-30] Palin et al reported that short- and medium-term water immersion could affect the properties of resin composites.[31] However, the effect of water immersion and temperature on the optical properties of composites, particularly fluorescence, has not been extensively discussed in the literature.

Single and double carbon bonds play an important role in the configuration of polymer backbones found in the organic matrix in resin-based materials and in organic compounds such as collagen in human dentin. Polymers can be subjected to degradation in an aqueous environment by 2 mechanisms: hydrolysis and enzymatic reactions.[32] This degradation occurs through oxidation, functional group attack, and chain cleavage.[33] Thus, the reduction of fluorescence found in this study could be explained by the degradation of organic complexes found in both dentin and composite resins. This could confirm the theory that luminophores are attached to the polymer chain by adding organic components with fluorescent features.[25] Mineral substances such as rare earth oxides have also been incorporated in polymer chains by chemical bonds that could equally be broken and leached by accelerated aging based on water immersion and temperature changes.[26]

A previous study reported that the fluorescence intensity of human dentin increased with age, temperature, and the length of time for which heat was applied.[34] Nevertheless, the physiological aging process in dental structures is a result of several factors that lead to the production of fluorescent substances over time and should not be compared with an artificial aging protocol. In the present study, the fluorescence of teeth was relatively constant after thermocycling. Temperature can act as a catalyst, speeding up photocleavage of organic bonds in natural teeth.[35] However, due to the short period of time of the thermocycling process in the present study, this effect was not observed in dental structure samples.

In enamel, fluorescence is attributed to the presence of organic compounds, which comprise <2% of its whole composition, resulting in reduced levels of fluorescence intensity. Due to these low fluorescence levels, some studies have investigated the optical properties of teeth using specimens made of dentin, or enamel and dentin separately.[3,15] In the present study, enamel and dentin were evaluated together in order to determine the fluorescence intensity of the tooth structure compared to enamel and dentin shades of composites overlapping, which was deemed more clinically relevant.

The 2 null hypotheses tested in the present study were rejected. Only 1 composite (Opallis) demonstrated similar fluorescence intensity when compared to tooth structure, and, with the exception of nanofilled composite Filtek Supreme, thermocycling had a negative effect on the fluorescence of all the evaluated composites.

The complete understanding of the optical properties of restorative materials and human teeth is imperative for the clinician to achieve a natural appearance of anterior esthetic dental restorations. Anterior teeth are frequently submitted to different types of illumination that are even more challenging. Additionally, manufacturers must use new technologies to develop composites with better optical properties that simulate those of dental tissues, and with increased stability against the adverse effects of the various conditions of the oral environment.

Conclusion

Within the limitations of this study, it can be concluded that the aging process carried out by thermocycling negatively affected the fluorescence intensity of all evaluated dental composites except Filtek Supreme, whereas the fluorescence of tooth structure was not affected. Also, among all the tested materials, Opallis was the only composite resin that showed similar fluorescence compared with the tooth structure before and after thermocycling.

Author information

Dr. Jablonski is in private practice in Curitiba, Brazil. Dr. Brum is a faculty member, Restorative Dentistry Department, School of Health and Biosciences, Pontificia Universidade Catolica do Parana, Curitiba, Brazil, where Dr. Takahashi is a postdoctoral candididate, and Drs. Rached and Souza are full professors, Graduate Program in Dentistry.

References

1. Lee YK, Lu H, Powers JM. Fluorescence of layered resin composites. *J Esthet Restor Dent.* 2005;17(2):93-100; discussion 101.
2. Dietschi D. Layering concepts in anterior composite restorations. *J Adhes Dent.* 2001;3(1):71-80.
3. Lee YK, Powers JM. Color difference of four esthetic restorative materials by the illuminant. *Am J Dent.* 2005;18(5):359-363.
4. Lee YK, Powers JM. Metameric effect between resin composite and dentin. *Dent Mater.* 2005;21(1):971-976.
5. Spitzer D, Bosch JJ. Total luminescence of bovine and human dental enamel. *Calcif Tissue Res.* 1976;20(2): 201-208.
6. Monsenego G, Burdairon G, Clerjaud B. Fluorescence of dental porcelain. *J Prosthet Dent.* 1993;69(1):106-113.
7. Magne P, Belser U. *Bonded Porcelain Restorations in the Anterior Dentition. A Biomimetic Approach.* Chicago: Quintessence Publishing; 2002:84-88.
8. Ecker GA, Moser JB, Wozniak WT, Brinsden GI. Effect of repeated firing on fluorescence of porcelain-fused-to-metal porcelains. *J Prosthet Dent.* 1985;54(2):207-214.
9. Vanini L. Light and color in anterior composite restorations. *Pract Periodontics Aesthet Dent.* 1996;8(7):673-682; quiz 684.
10. Peplinski DR, Wozniak WT, Moser JB. Spectral studies of new luminophors for dental porcelain. *J Dent Res.* 1982;59(9):1501-1506.
11. Wozniak WT, Moore BK. Luminescence spectra of dental porcelains. *J Dent Res.* 1978;57(11-12):971-974.
12. Baran GR, O'Brien WJ, Tien TY. Colored emission of rare earth ions in a potassium feldspar glass. *J Dent Res.* 1977;56(11):1323-1329.
13. Tani K, Watari F, Uo M, Morita M. Discrimination between composite resin and teeth using fluorescence properties. *Dent Mater J.* 2003;22(4):569-580.
14. Uo M, Okamoto M, Watari F, Tani K, Morita M, Shintani A. Rare earth oxide-containing fluorescent glass filler for composite resin. *Dent Mater J.* 2005;24(1): 49-52.
15. Takahashi MK, Vieira S, Rached RN, Almeida JB, Aguiar M, Souza EM. Fluorescence intensity of resin composites and dental tissues before and after accelerated aging: a comparative study. *Oper Dent.* 2008;33(2): 189-195.
16. Schulze KA, Marshall SJ, Gansky SA, Marshall GW. Color stability and hardness in dental composites after accelerated aging. *Dent Mater.* 2003;19(7):612-619.
17. Schulze KA, Tinschert J, Marshall SJ, Marshall GW. Spectroscopic analysis of polymer-ceramic dental composites after accelerated aging. *Int J Prosthodont.* 2003;16(4):355-361.
18. Powers JM, Fan PL, Raptis CN. Colors stability of new composite restorative materials under accelerated aging. *J Dental Res.* 1980;59(12):2071-2074.
19. Powers JM, Fan PL, Marcotte M. In vitro accelerated aging of composites and a sealant. *J Dent Res.* 1981; 60(9):1672-1677.
20. Lee YK, Lu H, Powers JM. Changes in opalescence and fluorescence properties of resin composites after accelerated aging. *Dent Mater.* 2006;22(7):653-660.
21. Lee YK, Lu H, Powers JM. Optical properties of four esthetic restorative materials after accelerated aging. *Am J Dent.* 2006;19(3):155-158.
22. The International Organization for Standardization. Determination of color stability. *ISO Standard 7491 Dental Materials.* 2nd ed. Geneva: 2000;1-4.
23. Lee YK, Lim BS, Rhee SH, Yang HC, Powers JM. Changes of optical properties of dental nano-filled resin composites after curing and thermocycling. *J Biomed Mater Res B Appl Biomater.* 2004;71(1):16-21.
24. Lee YK, Lim BS, Rhee SH, Yang HC, Powers JM. Color and translucency of A2 shade resin composites after curing, polishing and thermocycling. *Oper Dent.* 2005; 30(4):436-442.
25. Park MY, Lee YK, Lim BS. Influence of fluorescent whitening agent of the fluorescent emission of resin composites. *Dent Mater.* 2007;23(6):731-735.
26. Yan B, Wang QM. *In situ* composition and luminescence of terbium coordination polymers/PEMA hybrid thick films. *Opt Mater.* 2004;27:533-537.
27. Ferracane JL. Elution of leachable components from composites. *J Oral Rehabil.* 1994;21(4):441-452.
28. Calais JG, Soderholm KJ. Influence of filler type and water exposure on flexural strength of experimental composite resins. *J Dent Res.* 1998;67(5):836-840.
29. Oysaed H, Ruyter IE. Composites for use in posterior teeth: mechanical properties tested under dry and wet conditions. *J Biomed Mater Res.* 1986;20(2):261-271.
30. Beatty MW, Swartz ML, Moore BK, Phillips RW, Roberts TA. Effect of microfiller fraction and silane treatment on resin composite properties. *J Biomed Mater Res.* 1998;40(1):12-23.
31. Palin WM, Fleming GJ, Burke FJ, Marquis PM, Randall RC. The influence of short and medium-term water immersion on the hydrolytic stability of novel low-shrink dental composites. *Dent Mater.* 2005;21(9):852-863.
32. Lenz WR. Biodegradable polymers. *Adv Polym Sci.* 1993;107:1-40.
33. Ferracane JL. Hygroscopic and hydrolytic effects in dental polymer networks. *Dent Mater.* 2006;22(3): 211-222.
34. Matsumoto H, Kitamura S, Araki T. Autofluorescence in human dentine in relation to age, tooth type and temperature measured by nanosecond time-resolved fluorescence microscopy. *Arch Oral Biol.* 1999;44(4): 309-318.
35. March J. *Advanced Organic Chemistry: Reactions, Mechanisms and Structure.* 4th ed. Hoboken: John Wiley & Sons, Inc.; 1992:236-238.

Manufacturers

DENTSPLY Caulk, Milford, DE
800.532.2855, www.caulk.com

Extec Corp., Enfield, CT
800.543.9832, www.extec.com

FGM Produtos Odontologicos, Joinville, Brazil
55.47.3441.6100, www.fgm.ind.br

Heraeus Kulzer, South Bend, IN
800.435.1785, www.heraeus-dental-us.com

Hitachi High-Technologies Science America, Inc.,
Northridge, CA
818.280.0745, www.hitachi-hitec.com

Ivoclar Vivadent, Inc., Amherst, NY
800.533.6825, www.ivoclarvivadent.com

Kerr Corporation, Orange, CA
800.537.7123, www.kerrdental.com

Mitutoyo America Corporation, Aurora, IL
888.648.8869, www.mitutoyo.com

SPSS, Inc., Quarry Bay, Hong Kong
852.2811.9662, www.spss.com

Ultradent Products, Inc., South Jordan, UT
888.230.1420, www.ultradent.com