

Effects of thermocycling on the degree of cure of two lingual retainer composites

Bulent CATALBAS¹, Tancan UYSAL², Metin NUR³, Abdullah DEMIR⁴ and Beniz GUNDUZ⁵

¹Department of Orthodontics, Faculty of Dentistry, Kirikkale University, Kirikkale Universitesi, Dis Hekimligi Fakultesi, Kirikkale, Turkey.

²Department of Orthodontics, Faculty of Dentistry, Erciyes University, Erciyes Universitesi, Dis Hekimligi Fakultesi, Kayseri, Turkey.

³Department of Orthodontics, Faculty of Dentistry, Karadeniz Technical University, Karadeniz Teknik Universitesi, Dis Hekimligi Fakultesi, Trabzon, Turkey.

⁴Department of Orthodontics, Faculty of Dentistry, Selcuk University, Selcuk Universitesi, Dis Hekimligi Fakultesi, Konya, Turkey.

⁵Faculty of Chemistry, Selcuk University, Selcuk Universitesi, Fen-Edebiyat Fakültesi, Kimya Bölümü, Konya, Turkey.

Corresponding author, Tancan UYSAL; E-mail: tancanuysal@yahoo.com

The aim of this study was to evaluate the effects of thermocycling on the degree of cure (DC) and water sorption behavior of two lingual retainer composites. A total of 50 composite specimens, 5 mm diameter and 2 mm height, were prepared using Light Cure Retainer[®] (LCR) (Reliance) and Transbond Lingual Retainer[®] (TLR) (3M Unitek). After 40-second curing with a halogen light and after 24-hour water storage, the composite specimens were subjected to different thermocycling regimes. Absorbance peaks to monitor the DC were recorded using Fourier transform infrared (FTIR) spectroscopy, while water sorption was calculated in $\mu\text{g}/\text{mm}^3$. On DC, significant differences among the different thermocycling regimes were observed only for the TLR specimens ($p < 0.05$). On water sorption behavior, an increase in the number of thermal cycles resulted in increased water sorption for both composites, but the statistical differences in these groups were not significant. On the effect of thermocycling up to 20,000 cycles, present findings showed that LCR was less affected than TLR. Further, LCR exhibited higher DC and lower water sorption values than TLR after thermal cycling.

Keywords: Retainer, Degree of cure, Water sorption

Received Jun 3, 2009; Accepted Sep 17, 2009

INTRODUCTION

Photoactivated, resin-based composites (PARBCs) have become the material of choice for bonding lingual retainers today, as they offer ease of application and optimal handling characteristics to allow the clinician to shape and finish the adhesive around the lingual retainer wire for maximum patient comfort¹. Moreover, extended working time enhances accurate wire placement and minimizes product waste¹. While these excellent handling properties are both desirable and important, the degree of cure (DC) is also another very important factor for lingual retainer adhesives. This is because lingual retainers — unlike orthodontic brackets — are supposed to serve in the mouth for a long period of time². To achieve an optimum degree of cure for PARBCs, adequate light output and irradiation time are two indispensable requisites³. If these requisites are not fulfilled and incomplete curing occurs, these consequences await PARBCs: increase in water sorption, decrease in hardness, and softening of the polymer matrix caused by unreacted monomer which then led to deterioration in mechanical properties⁴.

For prolonged use in the oral cavity, the lingual retainer composites must possess certain physical properties to ensure clinical success⁵. On this note, DC has been reported to affect various properties of dental resin composites: wear resistance⁴, hardness, surface roughness, tensile and compressive strengths, and flexural modulus^{1,6,7}. It has been suggested that there is a correlation between DC and the resin formulation

of dental restorative resins⁸. According to one study² that investigated and compared the DC of two lingual retainer composites which were cured with fast halogen, plasma arc, and LED lights at different curing times, statistically significant differences were found between the DC values of the two lingual retainer composites cured with different light source-and-exposure time combinations. Then, for most investigations on orthodontic materials, the focus was on shear bond strength assessment and comparison between chemically-cured and light-cured materials.

In the oral cavity, orthodontic composites are also routinely exposed to thermal changes. Air temperature, humidity, and air velocity during breathing can alter resting mouth temperature⁹. Therefore, it is equally important to establish whether these changes would also affect the physical or mechanical properties of dental composites. On this note, Bishara *et al.*¹⁰ suggested that thermocycling should be part of the testing protocol for all new composites that await to be tested. Thermocycling is an *in vitro* process through which the test material and/or the tooth are subjected to large temperature extremes compatible with the oral cavity¹¹.

On the polymerization reaction of PARBCs, it has been reported to continue even after the end of light irradiation with DC showing a gradual increase after light exposure¹². However, to date, limited information is available concerning this phenomenon. Coupled with a lack of published studies on the influence of thermocycling on the DC of lingual retainer

composites¹³), the purpose of this study was to evaluate the effects of post-cure thermocycling (0, 5,000, 10,000, 15,000, and 20,000 thermal cycles) on DC and water sorption behavior of two lingual retainer composites cured with a halogen light unit.

The null hypothesis for this study was that different thermocycling regimes would affect the DC and water sorption behavior of lingual retainer composites.

MATERIALS AND METHODS

Curing and thermocycling of composite specimens

Two different lingual retainer composites were used in the present study: Light Cure Retainer (LCR) (Reliance, Itasca, Illinois, USA) and Transbond Lingual Retainer (TLR) (3M Unitek, Monrovia, California, USA). For each composite material, 25 specimens with a diameter of 5 mm and a height of 2 mm were prepared using a Teflon mold and covered between two microscope slides. All composite specimens were light-cured using a conventional halogen light unit (Hilux 350, Express Dental Products, Toronto, Canada; power density: 440 mW/cm²), with the 10-mm-diameter light tip irradiating one side of the material for 40 seconds as per the manufacturers' instructions.

All cured specimens were then placed in distilled water at 37°C for 24 hours. After 24 hours of water storage, the specimens were thermocycled according to the ISO 11405 recommendation¹⁴. Five specimens of each composite group underwent 0, 5,000, 10,000, 15,000 and 20,000 complete cycles in distilled water between 5°C and 55°C, with a dwell time of 30 seconds in each bath and a transfer time of 15 seconds between baths.

Measurement of the degree of cure (DC)

The infrared (IR) spectrum shows the DC from monomer to polymer. For this reason, the IR spectra of the composite specimens were measured using a Fourier transform IR (FTIR) spectrometer (1600 Series, Perkin-Elmer, Norwalk, Connecticut, USA) with the potassium bromide technique¹⁵.

After thermocycling, the specimens were pulverized into a fine powder with a mortar and pestle. Fifty micrograms of the ground powder was mixed with 5 mg of potassium bromide powder (Carlo Erba Reagenti, Rodano, Michigan, USA), and the absorbance peaks were recorded using the diffuse reflectance mode of FTIR spectroscopy. To measure the control spectra for each composite, the ground powder of five uncured specimens for each composite material was smeared onto thin potassium bromide disks.

DC was directly related to the decrease of 1637 cm⁻¹ absorption on the FTIR spectra as follows:

$$\%DC = ((A_0 - A_t)/A_0) \times 100$$

where A_0 is the absorption of the peak at 1637 cm⁻¹ when time is equal to zero and A_t is the absorption at

time t ¹⁶.

Water sorption measurement

Water sorption was measured according to the method described by Satou *et al.*¹⁷. The diameter and thickness of each specimen were measured, and the volume (V_0) calculated. Each specimen was stored at 37°C in a desiccator for 3 days until a constant weight (W_0) was obtained.

After the specimens were subjected to 0, 5,000, 10,000, 15,000 or 20,000 complete cycles in distilled water between 5°C and 55°C, they were removed from the water bath. Surface water was absorbed with a blotter paper until the surface was free from visual moisture. Upon reweighing, this weight was recorded as W_1 . Water sorption (W_{sp}) in $\mu\text{g}/\text{mm}^3$ was then calculated using the following equation:

$$W_{sp} = (W_1 - W_0)/V_0$$

Statistical analysis

All statistical analyses were performed using the Statistical Package for Social Sciences (SPSS for Windows 13.0, SPSS, Chicago, Illinois, USA) with $p < 0.05$ as the level of significance. Kruskal–Wallis and Mann–Whitney U tests were used to assess the statistical differences in DC and water sorption between composites under different thermocycling regimes. An independent sample t-test was used to statistically evaluate the differences between the combined overall DC and water sorption values of the two composites.

RESULTS

DC data

Table 1 shows the descriptive statistics and multiple comparisons of the DC values of the two lingual retainer composites obtained under five different thermocycling regimes. According to Kruskal–Wallis test, statistically significant differences were observed among the different thermocycling regimes for the TLR group ($p < 0.05$). Thus, the null hypothesis of this study could not be rejected.

In the LCR group, increase in the number of thermal cycles produced an increase in DC until 20,000 thermal cycles, but the differences in DC among the different thermocycling regimes for this group were not significant. The lowest DC value of 83.00% was achieved at 20,000 thermal cycles, whilst the highest value of 86.83% was achieved at 15,000 cycles.

In the TLR group, different effects were observed after thermocycling. The highest DC value of 74.67% was achieved at 20,000 cycles, whilst the DC values produced at 0–15,000 thermal cycles were similar and showed no statistically significant differences. Significant differences were thus found between the DC value at 20,000 thermal cycles and the other thermocycling regimes ($p < 0.05$).

Overall DC data for the LCR and TLR groups were

Table 1 Descriptive statistics and multiple comparisons of the DC values of two lingual retainer composites obtained under five different thermocycling regimes

Composite	Thermal Cycles	Sample Size	Mean DC (%)	Std. Deviation	Std. Error	Minimum	Maximum	<u>Kruskal-Wallis Test</u>	<u>Mann-Whitney U Test</u>			
								Significance	Thermal Cycles			
									5000	10000	15000	20000
Light Cure Retainer	0	5	83.46	9.81	9.90	81.00	86.00	NS	NS	NS	NS	NS
	5,000	5	84.50	9.41	9.71	78.00	92.00		NS	NS	NS	NS
	10,000	5	84.03	9.93	9.96	83.00	85.00			NS	NS	NS
	15,000	5	86.83	9.69	9.84	83.00	91.00					NS
	20,000	5	83.00	9.56	9.78	77.00	86.00					
Transbond Lingual Retainer	0	5	66.59	9.33	9.66	58.00	73.00	*	NS	NS	NS	*
	5,000	5	66.32	9.25	9.63	56.00	73.00			NS	NS	*
	10,000	5	67.31	8.29	9.14	42.00	79.00				NS	*
	15,000	5	66.99	9.14	9.57	59.00	78.00					*
	20,000	5	74.67	9.62	9.81	69.00	78.00					

Table 2 Descriptive statistics and statistical comparisons of the overall degree of cure for two lingual retainer composites

Composite Type		Sample Size	Mean DC (%)	Std. Deviation	Std. Error	Independent Samples <i>t</i> -test (<i>p</i>)
C=C	Light Cure Retainer	20	84.37	3.54	0.79	0.000
	Transbond Lingual Retainer	20	68.38	9.31	2.08	

Table 3 Descriptive statistics and multiple comparisons of the water sorption values of two lingual retainer composites obtained under five different thermocycling regimes

Composite	Thermal Cycles	Sample Size	Water Uptake ($\mu\text{g mm}^{-3}$)	Std. Deviation	Minimum	Maximum	<u>Kruskal-Wallis Test</u>
							Significance
Light Cure Retainer	0	5	0.94	0.51	1.68	2.39	NS
	5,000	5	2.07	0.52	1.58	2.44	
	10,000	5	2.25	0.56	1.72	2.65	
	15,000	5	2.35	0.41	1.89	2.67	
	20,000	5	2.39	0.58	1.89	2.66	
Transbond Lingual Retainer	0	5	1.91	0.90	3.56	5.57	NS
	5,000	5	2.40	0.79	3.70	5.41	
	10,000	5	4.01	0.85	3.57	5.42	
	15,000	5	4.21	0.94	3.80	5.51	
	20,000	5	4.30	0.91	3.90	5.69	

Table 4 Descriptive statistics and statistical comparisons of water sorption data for two lingual retainer composites

Composite Type		Sample Size	Water Uptake ($\mu\text{g mm}^{-3}$)	Std. Deviation	Independent Samples <i>t</i> -test (<i>p</i>)
Water Sorption	Light Cure Retainer	20	2.01	0.56	0.000
	Transbond Lingual Retainer	20	4.40	0.96	

acquired by pooling the values produced by the different thermocycling regimes. The overall data were subjected to statistical evaluation using an independent sample t-test. Results in Table 2 revealed that the overall DC value of TLR ($68.38 \pm 9.31\%$) was significantly lower ($p < 0.001$) than that of LCR ($84.37 \pm 3.51\%$).

Water sorption data

Table 3 presents the descriptive statistics and multiple comparisons of the water sorption values of the two lingual retainer composites obtained under five different thermocycling regimes. For both LCR and TLR, an increase in the number of thermal cycles resulted in increased water sorption, but the differences within these groups were not significant.

Water sorption data for the LCR and TLR groups were acquired by pooling the values produced by the different thermocycling regimes. Statistical analysis in Table 4 showed that the water sorption value of TLR ($4.40 \pm 0.96 \mu\text{g}/\text{mm}^3$) was significantly higher than that of LCR ($2.01 \pm 0.56 \mu\text{g}/\text{mm}^3$) ($p < 0.001$).

DISCUSSION

Effect on the DC of lingual retainer composites

One objective of this study was to evaluate and compare the effects of thermocycling in water on the DC of two different lingual retainer composites. Findings of this study revealed statistically significant differences among the DC values of the TLR composite under different thermocycling regimes. Thus, the null hypothesis could not be rejected. Besides, the overall DC values of both composites were also statistically different.

Several techniques have been introduced to determine the DC of materials. Amongst which, FTIR has been proven to be a powerful tool in material characterization^{2,6,18,19} as it detects the C=C stretching vibrations directly before and after the curing of materials. Although FTIR is widely used as a quick and reliable method to measure DC, it is not without limitations and shortcomings. In the present study, the DC values were average values obtained from a total of five composite specimens for each thermocycling regime and that the depth of cure was ignored. In other words, any possible DC differences between the top and bottom surfaces of the cured resin were not taken into consideration. Another limitation of this research was the relatively small number of specimens per test group ($n=5$), although many published studies had used smaller sample sizes^{2,6,17,20-22}.

According to many researchers that have used different indirect methods to evaluate the DC from monomer to polymer²³, the DC for restorative resins reportedly ranged from 50% to 80%. To the best of the authors' knowledge, only one report was found in published literature that investigated the DC of lingual retainer composites. Üşümez *et al.*² evaluated the DC of two different lingual retainer composites, LCR and TLR — which were the same as those evaluated in the

present study. The DC values determined by Üşümez *et al.*² for TLR and LCR were $55.5 \pm 10.3\%$ and $67.6 \pm 5.7\%$ respectively. In the present study, statistical evaluation of the pooled overall DC data of the same two composites yielded $68.38 \pm 9.31\%$ for TLR and $84.37 \pm 3.54\%$ for LCR.

Although the 68.38% DC obtained for the TLR composite was significantly lower than that of LCR, both DC values were higher than the previous reported DC range of 45–67% for set orthodontic resins²³. Moreover, these values were close to those reported by Eliades *et al.*¹⁸ for the different light-, chemical-, or dual-cured orthodontic adhesive resins. Therefore, it could be suggested that a favorable DC would likewise be obtained if PARBCs were used as lingual retainer adhesives. At this juncture, it must be clarified that on one hand, the type of composite used could affect the DC considerably²; on the other hand, DC has been found to affect the mechanical properties of composites. Nonetheless, no information is currently available on the effect of DC on the clinical performance of lingual retainer composites²³.

On the effect exerted by the type of composite used on DC, it is noteworthy that the two composite materials tested in this study differed in numerous aspects: viscosity, chemical structure of the dimethacrylate monomer, translucency, photoinitiator concentration, and color tone (shade). Consequently, the results obtained in this study reflected these differences^{2,16,24}. It has also been reported that monomers such as 2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane (Bis-GMA) and triethylene glycol dimethacrylate (TEGDMA) reached 85–90% of their final DC in 40 seconds with a QTH light unit¹³. In light of these findings, the implication is that if DC increases with greater light intensity and a longer irradiation time, then a stronger light source or a longer irradiation period in routine clinical practice could assuredly improve the curing of lingual retainer composites which are used to bond wire retainers on the lingual side of the teeth.

At this juncture, it must be put into perspective that the results of this *in vitro* study were obtained under well-controlled laboratory conditions. A host of variables — such as the presence of heat, daylight, and quality of resin infiltration — could affect DC and the resultant mechanical properties of the polymer created²⁵. Ramoglu *et al.*¹ investigated the effects of accelerated aging (different weather and light conditions) on the surface microhardness and roughness of two light-cured lingual retainer adhesives and found that accelerated aging significantly changed the mechanical properties of these tested materials. It was also likely that the varying results could be attributed to differences in resin and filler composition, extent of cure, and testing method. In light of these concerns and consequences, future investigations should take into consideration the effects of different physical conditions on the DC of resin-based composites.

Effect on the water sorption behavior of lingual retainer composites

Many dental materials are known to interact with components of the oral environment, such as heat and humidity. To determine the clinical efficacy of dental adhesive systems, laboratory tests such as the thermocycling test are often used to evaluate the performance of dental adhesive resins before proceeding with long-term clinical trials²⁶⁾. The thermocycling test is an *in vitro* process through which test specimens are subjected to temperature extremes using water baths in a bid to simulate conditions in the oral cavity¹¹⁾. For dental adhesive resins, their interaction with water causes the latter to diffuse into the matrix, resulting in both hygroscopic expansion and chemical degradation of the composite resin material²⁷⁾. As for the amount of water absorbed and the rate of absorption, they are diffusion-controlled²⁸⁾ and are dependent on several factors, many of which are material-dependent.

In the current study, the two lingual retainer composites tested did not demonstrate similar changes in DC after being subjected to thermal cycling in water. The DC value of TLR was increased while that of LCR was decreased after 20,000 thermal cycles. Changes in the DC property of a material might be related predominantly to the water uptake capacity of the tested material, and that degradation of a material after water sorption is indicated by an increase in C=C binding. Therefore, results of the present study showed that LCR was less affected than TLR composite after 20,000 cycles of thermocycling.

With PARBCs, water sorption is a critical property because of the manifold consequences brought about by the absorbed water: (1) increases the volume of the material²⁹⁾; (2) acts as a plasticizer and increases the deterioration of the resin matrix; and (3) affects the color stability of the composite material since water-soluble monomers can penetrate the outer border of the lingual retainers and lead to discoloration of the composite. According to Ferracane *et al.*³⁰⁾, the absorbed water causes the polymer network to soften by swelling and polymer swelling reduces the frictional forces between the polymer chains. It was also suggested that once the network is saturated with water and becomes softened, the composite structure stabilizes and there was no further reduction in properties within the time frame studied. This limited reduction in properties provided the evidence that further degradation, such as filler/matrix interfacial hydrolysis or polymer matrix crazing, may be absent or may not continue significantly once the material has become saturated and remains wet.

Another theory on water sorption is the osmotic pressure. It was suggested that acrylic polymers in a hydrogel matrix immersed in distilled water should absorb more water than those in saliva. Pertaining to this phenomenon, an investigation by Nicholson³¹⁾ showed results which were consistent with this theory, whereby there was greater equilibrium in water uptake in pure water than in salt solutions. Putting all the

theories and *in vitro* experimental results in perspective, it should be highlighted that clinical conditions may significantly differ from an *in vitro* setting. For this reason, our findings on TLR and LCR must be interpreted carefully. Nonetheless, the clinical significance of this study's findings was that the LCR composite exhibited lower water sorption than the TLR composite.

CONCLUSION

Within the limitations of an *in vitro* setting, findings of this study indicated that the LCR composite was less affected and thus more stable than the TLR composite under different thermocycling regimes. Comparatively, higher overall DC and lower water sorption values were achieved by the LCR composite.

REFERENCES

- 1) Ramoglu SI, Usumez S, Buyukyilmaz T. Accelerated aging effects on surface hardness and roughness of lingual retainer adhesives. *Angle Orthod* 2008; 78: 140-144.
- 2) Üşümez S, Büyükyilmaz T, Karaman AI, Gündüz B. Degree of conversion of two lingual retainer adhesives cured with different light sources. *Eur J Orthod* 2005; 27: 173-179.
- 3) Khalil SK, Allam MA, Tawfik WA. Use of FT-Raman spectroscopy to determine the degree of polymerization of dental composite resin cured with a new light source. *Eur J Dent* 2007; 1: 72-79.
- 4) Ferracane JL, Mitchem JC, Condon JR, Todd R. Wear and marginal breakdown of composites with various degrees of cure. *J Dent Res* 1997; 76: 1508-1516.
- 5) Usumez S, Buyukyilmaz T, Karaman A. Effect of a fast halogen and a plasma arc light on the surface hardness of orthodontic adhesives for lingual retainers. *Am J Orthod Dentofacial Orthop* 2003; 123: 641-648.
- 6) Rejman DJ, Eliades T, Bradley TG, Eliades G. Polymerization efficiency of glass-ionomer and resin adhesives under molar bands. *Angle Orthod* 2008; 78: 549-552.
- 7) Uysal T, Basciftci FA, Sener Y, Botsali MS, Demir A. Conventional and high intensity halogen light effects on water sorption and microhardness of orthodontic adhesives. *Angle Orthod* 2008; 78: 134-139.
- 8) Ferracane JL, Greener EH. The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins. *J Biomed Mater Res* 1986; 20: 121-131.
- 9) Gale MS, Darvell BW. Thermal cycling procedures for laboratory testing of dental restorations. *J Dent* 1999; 27: 89-99.
- 10) Bishara SE, Ajlouni R, Laffoon JF. Effect of thermocycling on the shear bond strength of a cyanoacrylate orthodontic adhesive. *Am J Orthod Dentofacial Orthop* 2003; 123: 21-24.
- 11) Helvatjoglu-Antoniades M, Koliniotou-Kubia E, Dionyssopoulos P. The effect of thermal cycling on the bovine dentine shear bond strength of current adhesive systems. *J Oral Rehabil* 2004; 31: 911-917.
- 12) Kildal KK, Ruyter IE. How different curing methods affect the degree of conversion of resin-based inlay/onlay materials. *Acta Odontol Scand* 1994; 52: 315-322.
- 13) Tarumi H, Imazato S, Ehara A, Kato S, Ebi N, Ebisu S. Post-irradiation polymerization of composites containing

- bis-GMA and TEGDMA. *Dent Mater* 1999; 15: 238-242.
- 14) International Organization for Standardization Technical Specification Report, ISO/TS 11405:2003.
- 15) Ferracane JL, Greener EH. Fourier transform infrared analysis of degree of polymerization in unfilled resin — Methods comparison. *J Dent Res* 1984; 63: 1093-1095.
- 16) Sideridou I, Tserki V, Papanastasiou G. Effect of chemical structure on degree of conversion in light-cured dimethacrylate-based dental resins. *Biomaterials* 2002; 23: 1819-1829.
- 17) Satou N, Matsumae I, Khan AM. Surface characteristics and staining of experimental visible light-cured unfilled resins. *J Mater Sci Technol* 1993; 4: 66-70.
- 18) Eliades T, Eliades G, Bradley TG, Watts DC. Degree of cure of orthodontic adhesives with various polymerization initiation modes. *Eur J Orthod* 2000; 22: 395-399.
- 19) Wendl B, Droschl H, Kern W. A comparative study of polymerization lamps to determine the degree of cure of composites using infrared spectroscopy. *Eur J Orthod* 2004; 26: 545-551.
- 20) Peutzfeldt A, Sahafi A, Asmussen E. Characterization of resin composites polymerized with plasma arc curing units. *Dent Mater J* 2000; 16: 330-336.
- 21) Silikas N, Eliades G, Watts DC. Light intensity effects on resin-composite degree of conversion and shrinkage strain. *Dent Mater J* 2000; 16: 292-296.
- 22) Caughman WF, Chan DCN, Rueggeberg FA. Curing potential of dual-polymerizable resin cements in simulated clinical situations. *J Prosthet Dent* 2001; 86: 101-106.
- 23) Kakaboura A, Eliades G, Palaghias G. An FTIR study on the setting mechanism of resin-modified glass ionomer restoratives. *Dent Mater* 1996; 12: 173-178.
- 24) Ferracane JL, Aday P, Matsumoto H, Marker VA. Relationship between shade and depth of cure for light-activated dental composite resins. *Dent Mater* 1986; 2: 80-84.
- 25) De Munck J, Van Meerbeek B, Yoshida Y, Inoue S, Vargas M, Suzuki K, Lambrechts P, Vanherle G. Four-year water degradation of total-etch adhesives bonded to dentin. *J Dent Res* 2003; 82: 136-140.
- 26) Hasegawa T, Retief DH, Russell CM, Denys FR. Shear bond strength and quantitative microleakage of a multipurpose dental adhesive system resin bonded to dentin. *J Prosthet Dent* 1995; 73: 432-438.
- 27) Yap AU, Wee KE. Effects of cyclic temperature changes on water sorption and solubility of composite restoratives. *Oper Dent* 2002; 27: 147-153.
- 28) Braden M, Causton EE, Clarke RL. Diffusion of water in composite filling materials. *J Dent Res* 1976; 55: 730-732.
- 29) Gigo DF, Franco EB, Mondelli RFL, Francisconi PAS, Navarro MFL. Diametral tensile strength and water sorption of glass-ionomer cements used in atraumatic restorative treatment. *J Appl Oral Sci* 2003; 11: 96-101.
- 30) Ferracane JL, Berge HX, Condon JR. *In vitro* aging of dental composites in water — effect of degree of conversion, filler volume, and filler/matrix coupling. *J Biomed Mater Res* 1998; 42: 465-472.
- 31) Nicholson JW. The physics of water sorption by resin-modified glass-ionomer dental cements. *J Mater Sci Mater Med* 1997; 8: 691-695.