*Bull Tokyo Dent Coll (2009) 50(4): 183–190*

**Original Article**

# Light Energy Transmission through Composite Influenced by Material Shades

**Ricardo Danil Guiraldo, Simonides Consani*, Rafael Leonardo Xediek Consani**, Sandrine Bittencourt Berger*, Wilson Batista Mendes and Mario Alexandre Coelho Sinhoreti***

*Prosthodontics Post-graduate Program, College Pythagoras, Belo Horizonte, MG, Brazil*
* *Department of Restorative Dentistry, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil*
** *Department of Prosthodontics and Periodontics, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil*

Received 6 April, 2009/Accepted for publication 14 September, 2009

## Abstract

This study investigated the influence of different composite resin shades on light energy transmission through the composite, hardness and cross-link density (CLD). The composite Filtek Z250 was used in shades A1, A2, A3, A3.5 and A4. A quartz tungsten halogen light curing unit was used at an irradiance of 900 mW/cm$^2$. Thirty specimens were made for each shade. Light energy transmission that passed through the composite was calculated ($n=10$). Differences in Knoop hardness between the top and bottom (DKH) of the same specimen were calculated ($n=10$). The Knoop hardness value for each surface was recorded as the average of three indentations ($KHN_1$). Thereafter, the specimens were soaked in absolute ethanol for 24 hr at room temperature, and hardness was again determined ($KHN_2$). The CLD was estimated by the softening effect produced by ethanol, *i.e.*, by decrease in hardness. The percentage of decrease in $KHN_2$ compared with $KHN_1$ (PD) in the same specimen was then calculated for both surfaces ($n=10$). The data were submitted to an ANOVA in different tests (Light energy transmission, Knoop hardness and CLD). The irradiance of light that passed through composite shade A1 (408 mW/cm$^2$) was statistically greater than that through shade A2 (376 mW/cm$^2$), and was greater through A2 than A3 (359 mW/cm$^2$) and through A3 than A3.5 (327 mW/cm$^2$); A3.5 showed no statistical difference when compared to A4 (324 mW/cm$^2$). The DKH of A4 (20.56%) was not statistically higher than that of A3.5 (20.14%), which was greater than that of A3 (14.08%), A2 (11.65%) and A1 (9.06%). There was no statistical difference in CLD. Darker shades had a significant influence on light energy transmission through dental resin composite and its hardness. However, CLD was not affected by darker dental composite shades.

Key words: Composite resin—Shades—Light energy transmission—Hardness—Cross-link density

## Introduction

A dental composite resin can be defined as a 3-dimensional combination of at least two chemically different materials, with a different interface separating the components. Basically, they are composed of an organic matrix, load particles (glass, quartz and/or melted silica) and a bonding agent, usually an organic silane, with a dual characteristic enabling chemical bonding with the load particle and co-polymerization with the monomers of the organic matrix[23].

Photo-activation is performed with visible light belonging to the blue area of the electromagnetic spectrum to excite camphorquinone (the most commonly used photo-initiator in composite resins), which has an absorption spectrum in the interval between 410 and 500 nm[8]. Among the photo-activation units available on the market, the most traditional ones are those that use quartz tungsten halogen light as light curing unit[17].

Currently, some composites, especially those of lighter colors, use photo-initiators or co-initiators as alternative systems, because camphorquinone (CQ) is yellow, which may compromise the esthetic properties of the composite[22]. The translucency of a resin composite may contribute to shade matching by allowing the surrounding tooth structure to shine though[25], and dental clinicians have frequently observed this "chameleon" effect of the resin composite[32]. The translucency of esthetic restorative materials has usually been determined with the translucency parameter[18], which is the different color of a uniform thickness of a specimen against a white and a black background and corresponds directly to common visual assessment of translucency[18].

The advantage of the Knoop hardness test of a composite is the correlation between the Knoop hardness and the degree of monomer conversion (DC)[9,13,27]. Mechanical properties of dental resin composites are directly influenced by DC[13]. Thus, after light curing, activation is desirable for this restorative material in order to attain the best mechanical properties to convert all of its monomers into a polymer. However, this has not been observed up to now and the polymerization rate is no higher than 61% on the surface directly illuminated by the light curing unit[33], and always presents a reduction of this polymerization rate as a function of the depth[24].

Nevertheless, although the DC is an important factor, it does not provide a complete characterization of the network structure, as polymers with a similar DC may present a distinct cross-link density (CLD) due to differences in the linearity of the chains[3,30]. In cross-link (CL) systems, residual double bonds in the polymer do not necessarily indicate free monomers, but may also result from pendant double bonds that are tied into the polymer network. CL is an important factor for good network formation and physical properties[16]. The extent of CL, pendant double bonds, and leachable components has been analyzed by the extraction of methyl methacrylate and CL dimethacrylate from denture base polymers[28]. Moreover, CLD has been indirectly assessed by polymer softening after exposure to ethanol[3,4].

The aim of study was to investigate the influence of different shades on the correlation between light energy transmission through the composite, hardness and CLD. It was hypothesized that light energy transmission through composite resin of different shades would influence hardness and CLD.

## Materials and Methods

The restorative resin composite (Filtek Z250; 3M-ESPE, St. Paul, MN, USA) used in this study comprised the following shades: A1, A2, A3, A3.5 and A4 (Table 1).

A quartz tungsten halogen (QTH) light curing unit (XL 2500; 3M-ESPE) was used at an irradiance of 900 mW/cm$^2$. The light curing unit (LCU) power (mW) was measured with a power meter (Ophir Optronics; Har-Hotzvim, Jerusalem, Israel). The tip diameter was measured with a digital caliper (digital caliper; model CD-15C; Mitutoyo, Kawasaki,

Table 1 Information on Filtek Z250 composite, used according to manufacturer's instructions

| Shade | | Organic matrix/Filler/Photo-initiator | | | Batch number |
|---|---|---|---|---|---|
| Hues | Values | | | | |
| A | 1 | BIS-GMA, UDMA and BIS-EMA | 60% by volume (mean of 0.19 to 3.3$\mu$m)–Zr and Si | Camphorquinone | 5UR |
| A | 2 | | | | 8GH |
| A | 3 | | | | 5CG |
| A | 3.5 | | | | 6HF |
| A | 4 | | | | 7TE |

Japan) to determine tip area. Irradiance was calculated by dividing light power by tip area. Spectral distributions were obtained using a spectrometer (USB 2000; Ocean Optics, Dunedin, FL, USA).

Standardized cylindrical specimens were obtained by placing the composite into a circular elastomer mold (2 mm in thickness $\times$7 mm in diameter). The bottom and top surfaces were covered with a transparent polyester strip and photo-activated by LCU. Photo-activation was performed for 20 sec, in accordance with the manufacturer's recommendations. For photo-activation, the curing tip was positioned close to the elastomer mold/restorative composite set. For each color, 30 specimens were made, measuring 2 mm in thickness by 7 mm in diameter.

**1. Light energy transmission test**

Ten specimens were randomly connected to the LCU tip with black adhesive paper. The light that passed through the composite was measured with a power meter (Ophir Optronics). Spectral distributions were obtained using a spectrometer (USB 2000). Irradiance data were submitted to an ANOVA and the means were compared by the Student's *t*-test ($\alpha$=0.05).

**2. Knoop hardness test**

After the photo-activation procedure, 10 randomly selected specimens were dry-stored at 37°C for 24 hr. Thereafter, both the top and bottom surfaces were polished with #200, 400 and 600 grit SiC abrasive (Carborundum; Saint-Gobain Abrasives, Recife, PE, Brazil) to obtain polished and flattened surfaces.

Indentations for Knoop hardness number (KHN) measurements were performed sequentially in a hardness tester machine (HMV 2; Shimadzu, Kyoto, Japan).

Three readings were taken on the top and bottom surfaces under a load of 50 gf for 15 sec. The KHN for each surface was recorded as the mean of three indentations.

After this, difference in Knoop hardness between the top and bottom (DKH) was calculated in each specimen. The DKH data were submitted to an ANOVA and the means were compared by the Student's *t*-test ($\alpha$=0.05).

**3. Cross-link density test**

After the photo-activation procedure, 10 randomly selected specimens were dry-stored at 37°C for 24 hr. Thereafter, both the top and bottom surfaces were polished with #200, 400 and 600 grit SiC abrasive (Carborundum) to obtain polished and flattened surfaces.

Indentations for Knoop hardness measurements were made sequentially in a hardness tester machine (HMV 2).

Three readings were taken on the top and bottom surfaces under a load of 50 gf for 15 sec. The Knoop hardness number for each surface was recorded as the average of three indentations ($KHN_1$). The specimens were soaked in absolute ethanol for 24 hr at room temperature to soften the material[30], and the hardness was again determined ($KHN_2$). The CLD was estimated by the softening effect produced by ethanol, *i.e.*, by decrease in hardness. The percentage of decrease in $KHN_2$ in comparison with $KHN_1$ (PD) in the same specimen was then calculated for both surfaces. The PD data were submitted to a two-way ANOVA (shade vs. surface) and the means were compared by the Tukey's test ($\alpha = 0.05$).

Table 2 Mean irradiance through Filtek Z250 composite

| Shade | Irradiance ($mW/cm^2$) |
|---|---|
| A1 | 408 (9) a |
| A2 | 376 (5) b |
| A3 | 359 (3) c |
| A3.5 | 327 (4) d |
| A4 | 324 (8) d |

Mean values followed by different lowercase letters differ statistically by Student's *t*-test at 5% level of significance. ( ) Standard Deviation.

Table 3 Mean values of difference in knoop hardness between the top and bottom surfaces (DKH)

| Shade | Knoop hardness number (KHN) | | DKH (%) |
|---|---|---|---|
| | Top | Bottom | |
| A1 | 72.4 (2.2) | 65.9 (2.9) | 9.06 (0.88) a |
| A2 | 73.3 (3.1) | 67.0 (2.2) | 11.65 (0.81) b |
| A3 | 72.9 (3.4) | 62.7 (1.9) | 14.08 (0.93) c |
| A3.5 | 71.8 (2.3) | 57.9 (2.7) | 20.14 (1.48) d |
| A4 | 73.1 (2.8) | 58.1 (3.2) | 20.56 (1.26) d |

Mean values followed by different lowercase letters differ statistically by Student's *t*-test at 5% level of significance. ( ) Standard Deviation.

Table 4 Mean percentage of decrease in $KHN_2$ and $KHN_1$ (PD) values in top and bottom surfaces

| Shade | PD (%) | |
|---|---|---|
| | Top | Bottom |
| A1 | 23.45 (3.34) a | 23.88 (3.79) a |
| A2 | 24.56 (3.71) a | 24.14 (3.64) a |
| A3 | 25.27 (3.48) a | 25.77 (3.41) a |
| A3.5 | 23.62 (2.06) a | 23.13 (2.72) a |
| A4 | 24.96 (2.78) a | 25.15 (2.04) a |

Mean values followed by different lowercase letters in each column differ statistically by Tukey's test at 5% level of significance. ( ) Standard Deviation.

## Results

As shown in Table 2 ($p = 0.001$), the irradiance of light that passed through composite shade A1 (408 $mW/cm^2$) was statistically greater than that through shade A2 (376 $mW/cm^2$), and was greater through A2 than A3 (359 $mW/cm^2$) and through A3 than A3.5 (327 $mW/cm^2$); A3.5 showed no statistical difference when compared to A4 (324 $mW/cm^2$).

Table 3 ($p = 0.001$) shows that the DKH of A4 (20.56%) was not statistically higher than that of A3.5 (20.14%), which was greater than that of A3 (14.08%), A2 (11.65%) and A1 (9.06%).

No statistically significant difference was observed in PD values for each shade/surface ($p = 0.89$, Table 4).

Figure 1 (12.02 by 15.55 cm) shows the wavelength distributions of the QTH light curing unit and the light that passed through the specimens of different shades of composite.

## Discussion

The hypothesis that light energy transmission through composite resin of different shades would influence hardness and CLD was partially accepted.

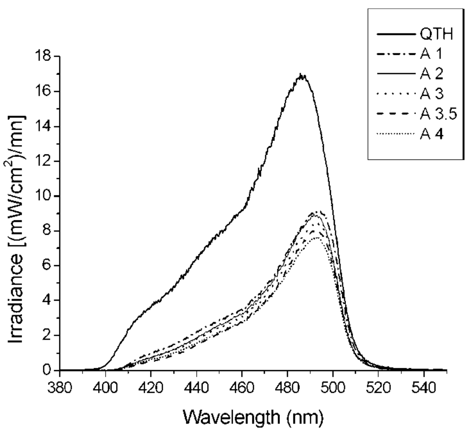


Fig. 1 Wavelength distributions of the QTH light curing unit and the light that passed through the specimens of different shades of the composite

Adequate polymerization is a crucial factor in obtaining an optimal physical mechanical performance of the dental resin composite[19]. Some previous studies have shown that factors such as filler and polymeric matrix refractive index, monomer type, filler type and filler content can influence the light transmittance of resin composite[6,12,21]. For this reason, it is possible to presume that loss of energy is mainly related to light absorption by the resin matrix and light dispersion by the composite resin filler. In the present study, the same resin composite (same polymeric matrix, filler particle type, percentage filler and photo-initiator) with different shades (same hues but different values) was used (Table 1).

During the photo-activation process, light that passes through a resin composite is absorbed and scattered[10]. Thus, light intensity is attenuated and its effectiveness reduced as depth increases[37,38]. Nevertheless, polymerization depth depends on light irradiance, exposure time and several other factors such as material composition[5], resin composite shade[35] and translucency[15]. Light energy transmission through darker shades is diminished due to opacity[29].

For Class II restorations, it is impossible to place the light guide directly on top of the composite restoration. The tooth structure or opaque matrix bands can shadow the light. Aged light sources do not provide optimal light energy output. In addition, placement of 2-mm thick composite increments may diminish light energy transmission. All of these factors tend to compromise the degree of polymerization of the restoration, which could result in diminishing its physical properties and decreasing its longevity[29]. In the current study, lighter shades produced statistically higher values than darker shades (Table 2), probably due to the tendency of composites of darker shades to absorb a greater amount of light than those of lighter shades[2].

The polymerization of light-cured composite resins starts and is sustained when the rate of delivery of photons from the LCU is sufficient to maintain the photo-initiator compound, CQ, in its excited or triplet state. In this state, CQ overreacts with an amine-reducing agent in order to form free radicals[7,26]. Resin shade is a factor that can alter polymerization efficacy[1]. As mentioned above, light energy transmission through darker shades is diminished due to opacity[29]. Opaque shades decrease the capacity of the light to penetrate the bulk of the resin composite[31]. In the current study, darker shades showed a higher DKH than lighter shades (Table 3), probably due to the lower rate of light that passed through the dental composite (Fig. 1).

Insufficiently polymerized composite resin may present quite a large number of problems such as poor color stability, greater stain uptake and risk of pulp aggression by non-polymerized monomers and portions of the material with different values of Young's modulus[31]. It has been reported that loading well-polymerized composite layers that have been placed on poorly polymerized layers can lead to the composite restoration bending inward and displacing, causing marginal fracture, open margins and cusp deflection[31]. However, it has been suggested that a specimen of resin composite is adequately polymerized when there is no more than a 20% difference between the maximum hardness at the top of the composite and the hardness at the bottom[11,20,34]. According to the results of present study, all shades met this criterion effectively after polymerizing the 2-mm thick specimens (Table 3).

The density of cross-linking can be calculated using infrared spectroscopy[36]. The cross-link density of polymer can also be indirectly assessed by measuring its glass transition temperature[12,34]. However, such evaluations require complex tests and special equipment. On the other hand, some studies have used the softening test as an alternative method of evaluation[3,30,39,40], in which measurement is based on repeated hardness tests after the samples have been softened in organic solvents. It is generally accepted that highly cross-linked polymers are more resistant to degradation and to solvent molecules diffusing within their structure[14].

It has been observed that CL dimethacrylate networks swell when exposed to solvents. This occurs because the forces of attraction between the polymer chains exceed the forces of attraction between solvent molecules and components of those chains[14]. Therefore, the solvent penetrates the resin matrix and expands the openings among chains[30]. This solvent penetration ability is related to the solubility parameter, which describes the ability of molecules to penetrate and dissolve another substance[14,30]. Differences in the solubility parameter between the polymer and the solvent will determine the extent of solvent uptake[30]. Subsequently, when there is a small difference between the solubility parameter of the solvent and the polymer itself, higher solvent uptake will occur[14,30]. The CLD plays a major role in the properties of a polymer, as highly cross-linked materials generally exhibit increased fracture strength and resistance to wear[14]. Polymers with a high CLD may be advantageous not only because they may present enhanced mechanical properties, but also by being less susceptible to softening by food substances and enzymatic attack[3]. In the current study, the PD showed no statistically significant difference, with proportional diminishment in CLD (Table 4). Since solvent uptake and swelling are directly related to CLD, a polymer with fewer cross-links is more sensitive to the plasticizing action of solvents[14]. As softening was proportional, the polymers of different shades probably shared similar properties which correlated with CLD.

Although this study was performed *in vitro*, the result can be considered clinically relevant. According to Shortall *et al.*[31], loading well-polymerized composite layers that are placed on poorly polymerized layers can lead to the composite restoration bending inward and displacing, causing marginal fracture, open margins and cusp deflection. The results of this study showed that light energy transmission through the composites with darker colors was reduced (Table 2) and that deeper composite layers had lower hardness values when compared with upper composite layers, and this would be reflected in clinical restorations (Table 3). Therefore, clinicians should increase exposure time in darker composites to obtain improved restorations. Moreover, CL is an important factor for good network formation and physical properties[16]. However, polymers with a high CLD may be advantageous not only because they may present enhanced mechanical properties, but also by being less susceptible to softening by food substances and enzymatic attack[3]. Thus, irradiance must be sufficient to form free radicals and form polymers with high CLD.

In summary, darker shades had a significant influence on light energy transmission through the dental resin composite and, consequently, on its hardness. This fact does not interfere in the final quality of the polymer. Thus, the CLD was not affected by darker shades of dental composites.

## Conclusion

1. Light energy transmission through composite was influenced by resin shade.
2. Hardness of composites was influenced by resin shade.
3. CLD of composites was not influenced by resin shade.

## References


1) Aguiar FH, Lazzari CR, Lima DA, Ambrosano GM, Lovadino JR (2005) Effect of light curing tip distance and resin shade on microhardness of a hybrid resin composite. Braz Oral Res 19: 302–306.
2) Arakawa H, Fujii K, Kanie T, Inoue K (1998) Light transmittance characteristic of light-cured composite resins. Dent Mater 14:405–411.
3) Asmussen E, Peutzfeldt A (2001) Influence of pulse-delay curing on softening of polymer structures. J Dent Res 80:1570–1573.
4) Asmussen E, Peutzfeldt A (2003) Influence of selected components on crosslink density in polymer structures. Eur J Oral Sci 109:282–285.
5) Atmadja G, Bryant RW (1990) Some factors influencing the depth of cure of visible light-activated composite resins. Aust Dent J 35: 213–218.

6) Campbell PM, Johnst WM, O'Brien WJ (1986) Light scattering and gloss of an experimental quartz-filled composite. J Dent Res 65:892–894.
7) Caughman WF, Rueggeberg FA, Curtis JW Jr (1995) Clinical guidelines for photocuring restorative resins. J Am Dent Assoc 126:1280–1286.
8) Cook WD (1982) Spectral distributions of dental photo-polymerization sources. J Dent Res 61:1436–1438.
9) DeWald JP, Ferracane JL (1987) A comparison of four modes of evaluating depth of cure of light-activated composites. J Dent Res 66: 727–730.
10) Dos Santos GB, Monte Alto RV, Filho HR, da Silva EM, Fellows CE (2008) Light transmission on dental resin composites. Dent Mater 24:571–576.
11) Dunn WJ, Bush AC (2002) A comparison of polymerization by light-emitting diode and halogen-based light-curing units. J Am Dent Assoc 133:335–341.
12) Emami N, Sjödahl M, Soderhölm K-JM (2005) How filler properties, filler fraction, sample thickness and light source affect light attenuation in particulate filled resin composites. Dent Mater 21:721–730.
13) Ferracane JL (1985) Correlation between hardness and degree conversion during the setting reaction of unfilled dental restorative resins. Dent Mater 1:11–14.
14) Ferracane JL (2006) Hygroscopic and hydrolytic effects in dental polymer networks. Dent Mater 22:211–222.
15) Ferracane JL, Aday P, Matsumura H, Atsuta M (1986) Relationship between shade and depth of cure for light-activated dental composite resins. Dent Mater 2:80–84.
16) Floyd CJ, Dickens SH (2006) Network structure of Bis-GMA- and UDMA-based resin systems. Dent Mater 22:1143–1149.
17) Guiraldo RD, Consani S, Lympius T, Schneider LFJ, Sinhoreti MA, Correr-Sobrinho L (2008) Influence of the light curing unit and thickness of residual dentin on generation of heat during composite photoactivation. J Oral Sci 50:137–142.
18) Johnston WM, Ma T, Kienle BH (1995) Translucency parameter of colorants for maxillofacial prostheses. Int J Prosthodont 8:79–86.
19) Knezevic A, Tarle Z, Meniga A, Sutalo J, Pichler G, Ristic M (2001) Degree of conversion and temperature rise during polymerization of composite resin samples with blue diodes. J Oral Rehabil 28:586–591.
20) Leonard DL, Charlton DG, Roberts HW, Cohen ME (2002) Polymerization efficiency of LED curing lights. J Esthet Restor Dent 14: 286–295.
21) Masotti AS, Onófrio AB, Conceição EN, Spohr AM (2007) UV-vis spectrophotometric direct transmittance analysis of composite resins. Dent Mater 23:724–730.
22) Park YJ, Chae KH, Rawls HR (1999) Development of a new photoinitiation system for dental light-cure composite resins. Dent Mater 15: 120–127.
23) Peutzfeldt A (1997) Resin composites in dentistry: the monomer systems. Eur J Oral Sci 105:97–116.
24) Pianelli C, Devaux J, Bebelman S, Leloup G (1999) The micro-Raman spectroscopy, a useful tool to determine the degree of conversion of light-activated composite resins. J Biomed Mater Res 48:675–681.
25) Powers JM, Dennison JB, Lepeak PJ (1978) Parameters that affect the color of direct restorative resin. J Dent Res 57:876–880.
26) Rueggeberg FA (1994) Effect of light intensity and exposure duration on cure of resin composite. Oper Dent 19:26–32.
27) Rueggeberg FA, Craig RG (1988) Correlation of parameters used to estimate monomer conversion in a light-cured composite. J Dent Res 67:932–937.
28) Ruyter IE, Oysaed H (1982) Conversion in denture base polymers. J Biomed Mater Res 16:741–754.
29) Sakaguchi RL, Douglas WH, Peters MC (1992) Curing light performance and polymerization of composite restorative materials. J Dent 20: 183–188.
30) Schneider LF, Moraes RR, Cavalcante LM, Sinhoreti MA, Correr-Sobrinho L, Consani S (2008) Cross-link density evaluation through softening tests: effect of ethanol concentration. Dent Mater 24:199–203.
31) Shortall AC, Wilson HJ, Harrington E (1995) Depth of cure of radiation-activated composite restoratives—influence of shade and opacity. J Oral Rehabil 22:337–342.
32) Sidhu SK, Ikeda T, Omata Y, Fujita M, Sano H (2006) Change of color and translucency by light curing in resin composites. Oper Dent 31:598–603.
33) Silikas N, Eliades G, Watts DC (2000) Light intensity effects on resin-composite degree of conversion and shrinkage strain. Dent Mater 16:292–296.
34) Soh MS, Yap AU, Siow KS (2003) Effectiveness of composite cure associated with different curing modes of LED lights. Oper Dent 28: 371–377.
35) Tanoue N, Koishi Y, Matsumura H, Atsuta M (2001) Curing depth of different shades of

a photo-actived prosthetic composite material. J Oral Rehabil 28:618–623.

36) Turssi CP, Ferracane JL, Vogel K (2005) Filler features and their effects on wear and degree of conversion of particulate dental resin composites. Biomaterials 26:4932–4937.

37) Vargas MA, Cobb DS, Schmit JL (1998) Polymerization of composite resins: argon laser vs conventional light. Oper Dent 23:87–93.

38) Watts DC, Amer O, Combe EC (1984) Characteristics of visible-light-activated composite systems. Br Dent J 156:209–215.

39) Witzel MF, Calheiros FC, Gonçalvez F, Kawano Y, Braga RR (2005) Influence of photoactivation method on conversion, mechanical properties, degradation in ethanol and contraction stress of resin-based materials. J Dent 33:773–779.

40) Yap AU, Soh MS, Han TT, Siow KS (2004) Influence of curing lights and modes on cross-link density of dental composites. Oper Dent 29:410–415.

*Reprint requests to:*

Dr. Ricardo Danil Guiraldo
Dental Materials,
Department of Restorative Dentistry,
Piracicaba Dental School,
Av. Limeira 901, 13414-903,
Piracicaba, São Paulo, Brazil
Tel: +55-19-2106-5345
Fax: +55-19-3421-5218
E-mail: rickdanil@fop.unicamp.br