

SURFACE HARDNESS OF DENTAL COMPOSITE PHOTOPOLYMERIZED WITH DIFFERENT LIGHT SOURCES AND POLYMERIZATION TIME

ABSTRACT

AIM: this in vitro study was evaluate the microhardness in dental composites photo activated in a distance of 8mm, what simulates the first composite increment in a restoration class II. MATERIAL **AND METHODS:** Thereunto, 45 specimens were confectioned using a nanopaticulate composite (Filtek Z350 - 3M Espe. The specimens were divided randomly in 9 groups (n=5). Each group differed in the photoactivation mode used (1600 mW/cm2 Bluephase 16i (BLP); 800 mW/cm2 Ultralume 5 (ULT); 500 mW/cm2 XL3000 (XL)) and in the temperature of composite in the moment of polymerization (23°C, 54°C or 60°C). After the preparation of specimens, they were submitted to the microhardness Knoop test (KHN) on the surfaces and bottom, and the results were charted and submitted to the statistical analysis. **RESULTS:** There were no significant statistical differences in relation to the photoactivation modes used for all the surfaces and temperatures tested. For ULT, there was no significant statistical hardness in relation to the temperature of the composite. For XL and BLP modes, the increase in the temperature (56°C and 60°C) provided a hardness rise in the composite on both surfaces; for XL the temperature 56°C did not differ from the control (23°C); for BLP the temperature 60°C did not differ from the control. **CONCLUSION**: For all the situations tested, the top surface presented greater hardness than the bottom.

ARECO, Igor Melki*
GIORGI, Maria Cecília Caldas**
CATELAN, Anderson**
AMBROSANO, Gláucia Maria Bovi***
LIMA, Débora Alves Nunes Leite****
BARON, Giselle Maria Marchi****

AGUIAR, Flávio Henrique Baggio****

KEYWORDS

Surface hardness. Composites resins. Photoactivation. Polymerization.

INTRODUCTION

The polymerization reaction of dental composite occurs by conversion of monomer molecules in a structure of cross-linked polymers.^{1,2} When the canphorquinone, molecule responsible to start the polymerization reaction absorbs a light photon (final unity of light energy) in a wave-length about 467 nm, an electron of this molecule is driven to a higher energy level becoming in an excited state.3 Therefore, canphorquinone collides with an amine and a free radical is formed. This radical may react with a carbon double link (C=C) of a monomer molecule starting the polymerization reaction,4 in which the monomers had the double carbon link broken in one or both extremity react with other monomers in the same situation and form polymer molecules.

The formation of polymer macromolecules is associated with the contraction of the composite polymerization. 1,2 The higher the intensity of light energy (quantum) used in the photoactivation process, more photons will react with canphorquinone molecules inside the resinous matrx of composite, increasing the conversion degree; in other words, the quantity of monomers converted in polymers. Thereupon, the quantity of light energy is the main factor to conversion degree of composite. 5

However, it has been verified that resinous material is not completely

polymerized, because contains a small quantity of residual monomers between the structures formed.^{6,7} As the conversion degree is related with physical properties of the composite,⁸ the quantity of monomers remaining is a codetermining of physical properties of the polymer resulting.⁶

There are several factors that may affect the quantity of light energy which the bottom and top surface of an increment receives, like type and size of the tip of the light curing unit, distance between the tip of the light curing unit and the composite surface, light intensity emitted by the light curing unit, the specificity of light emitted by the light curing unit, interaction between the light wave-length of the light curing unit and the starter agent of polymerization reaction, time of photoactivation, composition, color, opacity and composite thickness.^{4,9-13} Whether the composite increment does not receive total energy enough to an adequate polymerization reaction, several problems may appear determining the clinical failure of restoration. Among them we can cite: changes in physical properties, increase in pigmentation rate, increase in wear rate, increase in the cytotoxicity potential by presence of residual monomer, decrease of elasticity module, weak bond among tooth, adhesive and composite, and higher probability of collapse in tooth restoration interface. 2,4,12,14,15

Among the factors which may reduce the light intensity that achieves the composite, the only one which cannot be controlled by the dentist during the performance of the restoration of a deep cavity is the distance between the tip of the light curing unit and the surface of the composite increment. According to Prati et al. (1999),¹⁶ only 1mm o fair interjacent between the tip of the light curing unit and the composite surface reduces intensity of the light energy in approximately 10%.

In clinical situations when there are deep cavities, the distance between the first composite increment and the tip of the light curing unit is commonly higher than 8 mm, consequently it would reduces the light intensity that achieves the composite surface, decreasing the conversion degree and/or leading to the formation of polymers with linear structures. In both situations, the composite will present inferior physical properties, superficial and interface discoloration, and will result in weakness of restoration.¹⁷ When it is in contact with the oral environment, this not adequately polymerized composite can be solubilized, what accelerates the process of solubility of the adhesive and enable the marginal infiltration and secondary caries.6

Recently, studies have shown that the temperature increase of dental composite in the moment of photoactivation increases the conversion degree of composite, improving some physical properties, without change the increase of intra-pulpal temperature.¹⁸

The distance between the tip of curing device and material surface is one factor that cannot be controlled by the dentist during the restorative procedure in deep cavities, decreasing the light intensity that reaches on the material. Thus, a lower monomer conversion and/or a higher formation of linear polymers can occur, resulting on a restoration with poor physical properties.

The purpose of this study was to evaluate the microhardness of a composite resin at distance, simulating the first increment of composite restoration, using different light-curing units and pre-heating temperatures. Two null hypotheses were tested: there would be no difference between the different (1) light-curing units and (2) pre-heating temperatures on the Knoop microhardness of the composite tested.

MATERIAL AND METHODS

In this study, a nanofilled (Filtek Z350, shade A2; 3M ESPE, St. Paul, MN, USA) composite resin was used. Forty five cylindrical specimens (n = 5) were prepared in circular Teflon mold for each composite, with 6 mm in internal diameter and 2 mm thickness, held between two glass slabs, separated by Mylar matrix strips, and then pressed with a 500 g load. The cavity was filled with one increment and randomly polymerized according to experimental groups: three LCUs (Bluephase 16i,

Ultralume LED 5, and XL 3000) and three temperatures of pre-heating of the composite resin (23, 54, and 60 °C). The composite resin used was kept in an incubator (502, Fanem Ltda, Guarulhos, SP, Brazil) with respective pre-heating temperature during one hour before use. Polymerization was performed with tip of curing unit positioned at 8 mm distant from top surface of the sample (controlled by an electronic digital caliper). The LCUs used, irradiance, and radiant exposure are presented in table 1.

Each specimen was removed of the mold and dry stored in a lightproof vial at 37 °C for 24 h. After this period, the surface of each specimen was polished 1200-grit silicon carbide (SiC) grinding paper (CarbiMet 2 Abrasive Discs, Buehler, Lake Bluff, IL, USA).

Knoop hardness number (KHN) reading was measured on the top and bottom surfaces of each sample using a microhardness tester (FM; Future-Tech Corp, Tokyo, Japan) with a Knoop diamond indenter under 10-g load for 10 s. Eight indentations were made on each surface of the specimen, the first at 500 μ m of boundary and others 7 from this, distanced 200 μ m. The average of the 8 KHN values was calculated for each specimen.

The results of KHN on the top and bottom surface were submitted to two-way split-splot analysis of variance (ANOVA) and Tukey's test at the pre-set alpha of 0.05. The factors light-curing unit and temperature were considered in the

parcels and the factor surface (top and bottom surfaces) was considered in the sub-factor.

RESULTS

The light-curing units used, irradiance (mW/cm²), and radiant exposure (J/cm²) are presented in Table 1. The results obtained are presented in the Table 2. ANOVA showed significant difference for the interaction of factors light-curing unit and temperature (p < 0.0004). For ULT, there were no significant hardness statistical differences related to the composite temperature. For XL and BLP modes, the increase of temperature (56°C and 60°C) provided a hardness increase on both surfaces of the composite; for XL, the temperature 56°C did not differ from the control (23°C); and for BLP, the temperature 60°C also did not differ from the control. In all the situations tested, the top surface presented higher hardness than the bottom surface.

DISCUSSION

The appropriate polymerization is an important factor to obtain adequate physical properties of composite resins, and it is related with the better clinical performance. However, there are factor which may affect the quantity of light energy that top and bottom surfaces of a composite increment receives.⁹⁻¹³

Table 1 - Light-curing units used, irradiance (mW/cm²), and radiant exposure (J/cm²).

Light-curing unit		Irradiance at 0 mm	Irradiance at 8 mm	Radiant exposure at 0 mm	Radiant exposure at 8 mm
Bluephase 16i	LED 2 nd generation	1100	400	22	8
Ultralume 5	LED 3 rd generation	700	280	14	5.6
XL 3000	Halogen 1	450	200	9	4

Bluephase 16i: Vivadent, Bürs, Austria; Ultralume LED 5: Ultradent, South Jordan, UT, USA; XL 3000: 3M/ESPE, Grafenau, Germany. Irradiance was monitored by a radiometer (model 100; Demetron/Kerr, Danbury, CT, USA).

Table 2. Microhardness (Kg/mm2) means (S.D.) of the composite resins according to light-curing units, pre-heating temperatures, and surface analyzed.

Surface	Source	Temperature (°C)				
		23	56	60		
Тор	BLP	63,5 (20,6)* Ba	83,2 (22,4)* Aa	63,5 (31,9)* ABa		
	ULT	70,4 (9,7)* Aa	65,3 (11,8)* Aa	88,4 (34,8)* Aa		
	Xl	53,4 (17,5)* Ba	59,9 (15,9)*ABa	78,3 (42,6)* Aa		
Bottom	BLP	19,5 (5,4) Ba	38,3 (8,1) Aa	20,6 (5,1) ABa		
	ULT	35,8 (9,7) Aa	28,8 (8,3) Aa	34,0 (5,1) Aa		
	Xl	16,3 (8,4) Ba	22,4 (3,6) ABa	43,6 (15,5) Aa		

^{*}It differs of bottom surface (p < 0.0001). Distinct letters (capital in the row and lower in the column within of each composite) are statistically different (p \leq 0.05).

Among these factor, the radiant light incidence emitted by different devices for photoactivation and the photoactivation temperature were analyzed in this study. The results showed that only the factor temperature was able to increase the hardness of composites photoactivated at 8mm distance. For all the factors studied, the top surface presented higher hardness when compared to the bottom one.

The composite resin has as characteristic reducing the intensity of light

that passes by the body, decreasing the action of polymerization of the sample. The light penetrates in the sample body and decreases its polymerization effectiveness, changing significantly the values until the base of the sample. It demonstrated higher hardness in the sample on the top region both in different temperatures and with different types of sources used in the research.

In relation to the sources studied (specificity of light on the composite hardness) both halogen light and LEDs of 2nd and 3rd

generation did not present statistical differences. However, it was verified that different temperatures influenced statistically the composite hardness. The type of composite used may explain the absence of differences among the photoactivation modes, both on the base and on the top surface. This type of composite facilitates the penetration of light in the body sample, and level the photoactivation modes with different radiant incidences on the surfaces studied.¹⁹

In relation to the temperature increase, there were no hardness significant statistical differences for ULT. For XL and BLP, the temperature increase (56°C and 60°C) provided the hardness increase in the composite on the two surfaces; for XL, the temperature 56°C did not differ from the control (23°C); and for BLP, the temperature 60°C also did not differ from the control.

The temperature affects directly the behavior of the composite polyemerization. The increase of temperature reduces the viscosity of the resinous system and increase the mobility of radicals, facilitating the polymerization process. The temperatures chosen were selected following the results of work by Daronch et a. (2006).¹⁸

The initial purpose of this study was that the increase of temperature could improve the hardness of composites when the

photoactivation occurs in a distance of 8mm from the tip of the light curing unit used.

However, the increase of temperature did not increase the hardness for ULT; and improved for XL and BLP, but with different temperatures. For XL, the better temperature was 60°C; and for BLP, the better temperature was 56°C. Then, the increase of temperature and the better temperature depend on the photoactivation mode used. New studies should be performed in order to prove whether the increase of temperature improves the hardness and the conversion degree of resinous systems, according to the photoactivation system used.

CONCLUSION

Considering the methodology used and on the results obtained, it is possible conclude that: (1) for all the experimental conditions, the top surface showed higher values of hardness when compared with the bottom surface; (2) related to the light-curing units, the results did not demonstrate hardness statistical significant differences; (3) the increase of temperature and the better temperature to improve the superficial hardness depend on the photoactivation mode used.

ACKNOWLEDGEMENT

This study was supported by FAPESP (#08/01086-8).

REFERENCES

- 1. Friedl KH, Schmalz G, Hiller KA, Märkl A. Marginal adaptation of class V restorations with and without softstart-polymerization. Oper Dent. 2000;25:26-32.
- 2. Feilzer AJ, De Gee AJ, Davidson CL. Quantitative determination of stress reduction by flow in composite restorations. Dent Mater. 1990;6:167–171.
- 3. Lehninger AL. Princípios de Bioquímica. São Paulo: Savier S/A, 5 ed, 1991.
- 4. Price RB, Dérand T, Lonev RW, Andreou P. Effect of light source and specimen thickness on the surface hardness of resin composite. Am J Dent. 2002;15:47-53.
- 5. Abade PF, Zanra VN, Macchi RL. Effect of photopolymerization variables on composite hardness. J Prosthet Dent. 2001;86(6):632:635.
- 6. Asmussen E, Peutzfeldt A. Influence of pulse-delay curing on softening of polymer structures. J Dent Res. 2001;80(6):1570-1573.
- 7. Silikas N, Eliades G, Watts DC. Light intensity effects on resin-composite degree of conversion and shrinkage strain. Dent Mater. 2000;16(4):292-296.
- 8. Rueggeberg FA, Caughman WF, Curtis Jr JW. Effect of light intensity and exposure duration on cure of resin composite. Oper Dent. 1994;19:26-32.
- 9. Shortall AC, Wilson HJ, Harrington E. Depth of cure of radiation-activated composite restoratives Influence of shade and opacity. J Oral Rehabil. 1995;22:337-342.
- 10. Correr Sobrinho L, Lima AA, Consani S, Sinhoreti MAC, Knowles JC. Influence of curing tip distance on composite Knoop hardness values. Braz Dent J. 2000;11(1):11-17.
- 11. Correr Sobrinho L, De Góes MF, Consani S, Sinhoreti MAC, Knowles JC. Correlation between light intensity

- and exposure time on the hardness of composite resin. J Mater Sci. 2000;11:361-364.
- 12. Yap AUJ. Effectiveness of polymerization in composite restoratives claiming bulk placement: impact of cavity depth and exposure time. Oper Dent. 2000;25:113-120.
- 13. Leloup G, Holvoet PE, Bebelman S, Devaux J. Raman scattering determination on the depth of cure of light-activated composites: influence of different clinically relevant parameters. J Oral Rehabil. 2002;29:510-515.
- 14. Ferracane JL, Greener EH. Fourier transform infrared analysis of degree of polymerization of unfilled resins: methods comparison. J Dent Res. 1984;63:1093.
- 15. Asmussen E, Peutzfeldt A. Influence of composition on rate of polymerization contraction of light-curing resin composites. Acta Odontol Scand. 2002;60:146-150.
- 16. Prati C, Chersoni S, Montebugnoli L, Montanari G. Effect of the air, dentin and resin- based composite thickness on light intensity reduction. Am J Dent. 1999;12(5):231-234.
- 17. Atmadja G, Bryant RW. Some factors influencing the depth of cure of visible light- ativated composite resin. Aust Dent J. 1990;35(3):213-218.
- 18. Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. J Dent Res. 2005 Jul;84(7):663-7.
- 19. Pablino, Cáceres Benitez. Evaluation of the effect of the temperature on the microhardness of a composite resin light-cured with halogen lamp and LED's. Araraquara, 2004.