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Effect of Light Energy Density on Conversion Degree and Hardness of Dual-cured Resin Cement

PCP Komori • AB Paula • AA Martin
RN Tango • MAC Sinhoreti • L Correr-Sobrinho

Clinical Relevance

Light energy density can influence the curing of dual-cured resin cement. The ultimate physical properties of dual-cured resin cement depend on light energy delivered from the light-curing unit. It can guide the clinicians to select the appropriate curing unit for curing dual cement.

*Paula Carolina de Paiva Komori, DDS, PhD, professor, Dental Materials and Prosthodontics Department, Sao Jose dos Campos Dental School–UNESP, Sao Jose dos Campos, SP, Brazil

Andréia Bolzan de Paula, PhD student, Piracicaba Dental School–University of Campinas, Dental Materials, São Paulo, Brazil

Airton Abrão Martin, PhD, Vale do Paraiba University, Research and Development Institute, São Paulo, Brazil

Rubens Nisie Tango, DDS, PhD, Sao Jose dos Campos Dental School–UNESP, Dental Materials and Prosthodontics, São Paulo, Brazil

Mario Alexandre Coelho Sinhoreti, DDS, PhD, Piracicaba Dental School–University of Campinas, Restorative Dentistry, São Paulo, Brazil

Lourenço Correr-Sobrinho, Piracicaba, Brazil

*Reprint request: Av Francisco José Longo 777 Jardim São Dimas, São José dos Campos, São Paulo 12245-000, Brazil; e-mail: komori@fosjc.unesp.br

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SUMMARY

This study evaluated the effect of different light energy densities on conversion degree (CD) and Knoop hardness number (KHN) of RelyX ARC (RLX) resin cement. After manipulation according to the manufacturer's instructions, RLX was inserted into a rubber mold (0.8 mm X 5 mm) and covered with a Mylar strip. The tip of the light-curing unit (LCU) was positioned in contact with the Mylar surface. Quartz-tungsten-halogen (QTH) and light-emitting diode (LED) LCUs with light densities of 10, 20 and 30 J/cm² were used to light-cure the specimens. After light curing, the specimens were stored dry in lightproof containers at 37°C. After 24 hours, the CD was analyzed by FT-Raman and, after an additional 24-hours, samples were submitted to Knoop hardness testing. The data of the CD (%) and KHN were submitted to two-way ANOVA and the Tukey's test (α=0.05). QTH and LED were effec-

tive light curing units. For QTH, there were no differences among the light energy densities for CD or KHN. For LED, there was a significant reduction in CD with the light energy density set at 10 J/cm². KHN was not influenced by the light-curing unit and by its light energy density.

INTRODUCTION

The resin-luting agent became popular because of its high strength, low solubility and esthetic appearance.[1] An increasing number of products are available on the market today. The majority of resin-luting materials employ dual-cured polymerization, which combines the desirable properties of both chemically-cured and light-cured cements, such as a wide range of shades, rapid initial hardening and further curing by the chemical catalyst[2] and extended working time, providing the operator with more control over setting the material than the chemically-cured system, alone.[3] On a two-paste system, the dual-cured resin-luting agents have both initiation systems, because the base paste usually contains camphorquinone, both aliphatic amine and aromatic tertiary amine and the catalyst paste, which contains benzoyl peroxide.[4]

Several studies investigated the effect of the polymerization mode on the degree of conversion, mechanical properties or the bond strength of resin-luting agents.[4-6] The manufacturers of dual-cured resin cements strive for these materials to be effectively polymerized solely by chemical reaction and the chemical curing to be recommended for cementation of the restorations where light penetration is compromised.[4] However, the majority of dual-cured resin cement does not reach similar conversion degree or hardness values when tested in the presence or absence of light activation.[4]

The activation of current resin-based materials by blue light is well established, but a problem arises as the intensity of the activating light is reduced by passage through air, tooth and restoration.[7] The most commonly used light-curing units are quartz tungsten halogen light-curing units that emit a wide spectrum of wavelengths.[8-9] These types of light sources usually operate at light intensities between 400 and 800 mW/cm² and polymerize composite within 40 seconds at depths of up to 2 mm.[10] Newly developed light curing units operate at relatively high intensity and are purposed to provide optimum properties to composites in a shorter exposure time.[9]

The hypothesis of the current study was that the conversion degree and hardness of dual-cured light activated resin cement is dependent on the light energy densities and light-curing unit.

METHODS AND MATERIALS

Specimen Preparation

The dual-cured resin cement used in the specimen preparation was RelyX ARC–RLX (3M ESPE, St Paul, MN, USA). Rubber molds 5 mm in diameter and 0.8 mm thick were used as matrix for the resin cement specimens. After manipulation according to the manufacturer's instructions, the cement was inserted into the mold and covered with a Mylar strip (Polidental Ind and Com, São Paulo, Brazil). The tip of the light unit was positioned in contact with the Mylar surface during light curing using a conventional quartz-tungsten-halogen light-curing unit, Degulux—QTH (Degussa Dental, Hanau, Germany) with a light intensity of 800 mW/cm² and a light emitting diode, Smartlite PS–LED (Dentsply, Konstanz, Germany) with a light intensity of 1100 mW/cm². The output power energy (mW) emitted by each light-curing unit was measured with a digital power meter (Ophir Optronics Inc, Danvers, MA, USA) and the diameter of the light guide tip (cm) was measured with a digital caliper (Mitutoyo Tokyo, Japan). The irradiance (mW/cm²) was computed as the ratio of the output power by the area of the light guide tip. Light densities of 10, 20 and 30 J/cm² were used to light-cure the specimens, which were obtained as the product of irradiance and time of exposure. After light curing, the specimens (n=5) were stored dry in lightproof containers at 37°C. After 24 hours, the top surface of each specimen was submitted to conversion degree analysis by FT-Raman. After an additional 24 hours, these same specimens were submitted to the Knoop hardness test.

Conversion Degree (CD)

The conversion degree was analyzed 24 hours after light curing. Spectra of the uncured and cured resins were obtained using an FT-Raman Spectrometer (RFS 100/S—Bruker Inc, Karlsruhe, Germany). To excite the spectra, the defocused λ 1064.1 nm line of an Nd:YAG laser source was used. The maximum laser power incident on the sample surface was about 200 mW and the spectrum resolution was 4^{cm-1}. The aluminum mold with uncured resin was positioned in the sample compartment and the sample stage was mounted on an optical rail. The FT-Raman spectra of the uncured resin were obtained using 200 scans without removing the resin of the aluminum rods. For each specimen surface, three spectra were acquired in three distinct points, obtaining 90 spectra in the total. The FT-Raman spectra were analyzed by selecting a spectra region from 1590 to 1660^{cm-1}. The Raman vibration stretching mode in 1609 and 1638^{cm-1} was fitted by Lorentzian shapes to obtain the height of the peaks using the Microcal Origin software. To calculate the conversion degree, the height ratio of the peaks at 1609 and 1638^{cm-1} were used in the Equation 1. The mean value and standard deviation were calculated for each series.

Equation 1:

DC (%) = 100*[1 – R cured/R uncured]

where R = band height at 1638^{cm-1}/band height at 1609^{cm-1}

The FT-Raman results were obtained for each specimen and the values were submitted to two-way ANOVA and the Tukey's test (α=0.05).

Knoop Hardness Number (KHN)

After 48 hours of light-curing, the specimens were longitudinally sectioned in two equal parts under water cooling with a diamond saw (Extec model 12205, Extec Corp, Enfield, USA). The exposed surfaces were sequentially polished under water cooling with #400, #600 and #1200 grit Silicon Carbide sandpapers (Carborundum, Saint-Gobain, Recife, Pernambuco, Brazil) for 15, 30 and 60 seconds, respectively, in a universal polishing machine model APL-4 (Arotec, Cotia, Brazil).

Microhardness measurements were performed in a Microhardness Tester model HMV-2 Shimadzu (Shimadzu, Tokyo, Japan). Three sequences of three indentations each (50g during 15 seconds) were performed to obtain three hardness values for each depth: 50 µm, 400 µm and 750 µm.

A mean hardness value was obtained for each specimen and the values were submitted to two-way ANOVA and to the Tukey's test (α=0.05).

RESULTS

The two-way ANOVA for CD is presented in Table 1 and shows statistical significance for light energy density (p=0.01) and the interaction "density" X "curing unit" (p=0.003). There was no statistically significant difference for the light curing unit (p=0.67).

For comparisons among the groups, the data of the CD was submitted to the Tukey's test (Table 2). For QTH, there was no significant difference among the different light energy densities, but for LED, there was a significant decrease in CD, with densities of 10 J/cm^2 (p<0.05).

Two-way ANOVA for KHN is presented in Table 3, which showed significance for curing unit (p=0.008) and interaction "density" X "curing unit" (p=0.003). There was no statistical significant difference in density (p=0.12).

In Table 4, the comparison among the groups can be verified. Both light-curing units (QTH/LED) presented statistically similar KHN mean values with different light energy densities.

Table 1: *Results of Two-way ANOVA (dependent variable: CD)*

Source of Variation	Df	Mean Square	F	*P*-value
Curing unit	1	0.37	0.17	0.67
Density	3	7.9	3.8	0.01*
Curing unit X Density	3	11.63	5.54	0.003*
Error	32	2.09		
Total	39	126.32		

**Statistically significant difference.*

Table 2: *Mean Values and Standard Deviations of Conversion Degree (%) for RLX in According to Light Energy Density and Time of Exposure (seconds)*

Density	10 J/cm^2 (seconds)	20 J/cm^2 (seconds)	30 J/cm^2 (seconds)
QTH	82.97(1.04)a/A (12 seconds)	83.88(2.82)a/A (25 seconds)	84.19(1.01)a/A (37 seconds)
LED	81.10(1.32)b/A (9 seconds)	83.60(1.35)a/A (18 seconds)	83.89(0.97)a/A (27 seconds)

**Means followed by distinct small letters in the same line and capital letters in the same column were statistically different (Tukey's test, p<0.05).*

Table 3: *Results of Two-way ANOVA (dependent variable: KHN)*

Source of Variation	Df	Mean Square	F	*P*-value
Curing unit	1	94.27	7.70	0.008*
Density	3	25.29	2.06	0.12
Curing unit X Density	3	71.01	5.80	0.003*
Error	32	12.23		
Total	39	774.76		

**Statistically significant difference.*

Table 4: *Mean Values and Standard Deviations of Conversion Degree (%) for RLX According to Light Energy Density and Time of Exposure (seconds)*

Density	10 J/cm² (seconds)	20 J/cm² (seconds)	30 J/cm² (seconds)
QTH	49.75(4.23)a/A (12 seconds)	48.62(2.81)a/A (25 seconds)	50.28(3.55)a/A (37 seconds)
LED	49.52(3.10)a/A (9 seconds)	50.89(2.05)a/A (18 seconds)	45.87(4.77)a/A (27 seconds)

**Means followed by distinct small letters in the same line and capital letters in the same column were statistically different (Tukey's test, p<0.05).*

DISCUSSION

Several studies measured the intensity of light-curing units and demonstrated that the degree of polymerization of resin cements decreased with the decrease in light intensity.[4-5] For resin composites, a light intensity up to 400mW/cm² is generally recommended.[11]

The results of the current study show that QTH provides the same resin cement values of CD and KHN when different light energy densities are compared. Cross-linking reaction and polymerization continue, even after light curing has been completed.[2] The light energy density promoted by this kind of light curing unit has the ability to activate polymerization and achieve reliable CD and KHN values. Both were measured in the same samples, the former 24 hours after light curing and the latter after an additional 24 hours, in an attempt to verify correlation between CD and KHN. According to the results, there is no correlation between these two variables. Under low light intensity irradiation, dual-cured resin cement still has a large amount of free radicals, mostly from chemical catalysts trapped in the hardening resin matrix. Although these trapped free radicals did not contribute significantly to the overall DC, they could improve the cross-linking density of dual-cured resin-luting agents by combining with a double link of methacrylate groups in local resin matrix to complete termination.[12]

LED presented a significant reduction in CD with 10 J/cm². It could result in high light intensity (1100 mW/cm²) combined with a short exposure time of 10 seconds. High intensity may produce more starter radicals and shorter polymer chains than a low intensity light curing unit.[13-14] Some dual-cured resin cements exposed to initial light curing polymerize fast, resulting in a viscous gel. The rapid increase in viscosity may hinder the migration of active radical components responsible for further chemically induced polymerization.[6]

RLX received a total of 11 J/cm², but several studies showed that at least 12 J/cm² is necessary for light curable resin composite-based materials to achieve reliable CD and KHN values.[15] The CD mean value was 81.10% and could not be correlated to KHN. Previous studies verified similar values of conversion degree in resin composite samples with different hardness values, which were attributed to differences in the polymer crosslinking density.[16] The ultimate hardness value of dual-cured resin cements depends on the amount of time exposure to the curing light.[5]

Most dual-cured resin cements still require photoactivation and demonstrate inferior hardness when light activation is omitted.[17] With a high-power light unit, more photons are available per second for absorption,[18] more photoinitiator reacts with amine and more free radicals are available for polymerization.[19] This photoinitiator is excited in the presence of light with an adequate wavelength and sufficient irradiance.[20] Rapid polymerization may also result in the formation of high cross-linked short polymer chains.[21]

The results of the current study show that all light energy densities achieve high CD and KHN values. Additionally, the light curing unit tip was directly in contact with the resin cement. In the dental practice, this only occurs with the cement margin of the restoration. Therefore, many other factors can modify this result, such as restoration shade, thickness and material type and commercial brands of material for indirect restoration. Further research and controlled clinical trials are needed before clinical protocol recommendations can be given.

CONCLUSIONS

For the dual-cured resin cement used:

1) QTH and LED were effective light curing units;
2) LED provided a significant reduction in CD, with a light energy density of 10 J/cm²;
3) KHN was not influenced by the light-curing unit or by its light energy density.

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