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Oral Sciences

# Polymerization of a dual-cured cement through Empress 2 porcelain: LED curing light vs. halogen curing unit

LOPES, Lawrence Gonzaga, FRANCO, Eduardo Batista, NAME-NETO, Abrão, HERRERA, Francyle S., MONDELLI, Rafael F. Lia, and PEDROSA, Sérgio de Freitas. **Polymerization of a dual-cured cement through Empress 2 porcelain: LED curing light vs. halogen curing unit.** Oral Sci., may/aug. 2005, vol.1, no.1, p.15-21.

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ABSTRACT - The aim of this study was to investigate the influence of light source, a LED unit and a halogen lamp (HL) on the effectiveness of Enforce dual-cured cement cured under a ceramic disc (IPS Empress 2). Three exposure times (60, 80, and 120 s) were also evaluated. Two experimental groups, in which the polymerization of the dual-cement was performed through a ceramic disc, and two control groups, in which the polymerization of the dual-cement was performed directly without the presence of a ceramic disc were subdivided into three subgroups (three different exposure times), with five samples in each: G1A- LED 60 s; G1B- LED 80 s; G1C- LED 120 s; G2A- HL 60 s; G2B - HL 80 s; G2C- HL 120 s; and control groups (photopolymerization without porcelain): G3A- LED 60 s; G3B- LED 80 s; G3C- LED 120 s; G4A- HL 60 s; G4B- HL 80 s, and G4C- HL 120 s. The Vickers hardness test was used. A four-way ANOVA and Tukey test ($p \leq 0.05$) were performed. In top surfaces, all the samples cured by LED have hardness values comparable with the samples cured by halogen lamp. In bottom surfaces, there were significant differences between light sources and exposure times, where 60 s of exposure time with LED was statistically inferior to that of halogen groups. Nevertheless, LED at 120 s was statistically similar to halogen at both 60 s and 80 s. LED technology is thus a possible method for photo-curing through Empress 2 restorations, however, it is necessary to increase the exposure time for thick cement layers.

KEYWORDS - Resin cement, microhardness test, dental ceramic, light emitting diode

Received September 18, 2004
Accepted December 10, 2004

## Introduction

Dental porcelains are widely used in aesthetic dentistry for veneers, crowns, inlays, and onlays. Aesthetics, strength, and clinical long life are the objectives of indirect restorations using ceramic systems. The great development in the area of dental porcelains has been due to the advancement of bonding procedures, which has led to an increase in the number of indirect restorations being performed and to the development of new treatment alternatives (1,2).

In general these restorations are cemented with a dual-cure resin cement that possesses photo and chemical curing components. Polymerization of the resin cement is related to the success of these types of indirect restorations, since the bond strength between porcelain and cement, and between cement and enamel and dentin will be dependent on achieving adequate polymerization of the resin cement. Thus, it is desirable for a resin cement to achieve a high degree of conversion under these indirect restorations, since the extent of cure of the resin cement affects the mechanical properties, solubility and biocompatibility (3,4).

The halogen bulb light curing units (LCUs) are most commonly used to cure resin materials (5). However, this technology has several drawbacks. Halogen

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bulbs have a limited effective lifetime of approximately 50-100 hours (6,7). These units provide a wide spectral output and require a dielectric pass-band filter, which removes the undesirable wavelengths, under 400 nm and over 500 nm (5,8,9). A LCU bulb, reflector, and filter degrade over time due to the high temperatures produced, leading to a reduction in light output. The result is a decrease in the LCU's effectiveness to cure dental composites (6,10).

A recent alternative to the halogen lamp is the light emitting diode (LED). In the area of dentistry, this technology was first mentioned in 1995 by Mills *et al*. (11). LED units emit visible blue light in a narrow bandwidth (450-490 nm) that corresponds closely to the absorption peak of camphoroquinone, the most commonly used photoinitiator in light-activated materials (12,13). Thus, filter systems are not required and heat is not generated (10,12-14). Additional advantages of using LED would be that they have a long lifetime, are low-cost, and are resistant to shock and vibration (10,13,14).

A recent series of papers have indicated that LED units are a viable light source for photopolymerization of direct resin restorations, especially with an increment thickness of about 2 mm (10-13,15). However, when general practitioners buy a light-curing unit, they will most probably use it for both direct and indirect restorations. Thus, studies that simulate conditions where the light must pass through porcelain to activate the resin cement are very important, since indirect porcelain restorations are considered a common clinical procedure in modern operative dentistry. The objective of this study was to evaluate the hardness of a dual-cured resin cement under a porcelain disc (IPS Empress 2) after photopolymerization with LED light curing unit (LCU) and conventional halogen LCU at three exposure times (60, 80, and 120 s).

## Materials and methods

The materials and curing units used in this study are shown in Table 1. This study evaluated the hardness of Enforce dual-cured resin cement when photocured through a porcelain disc (Empress 2- pressable porcelain) by LED or halogen LCU. Two experimental and two control groups were established (Table 2). Each group was subdivided into three subgroups in accordance with three exposure times (60, 80, and 120s) commonly used and recommended for polymerization of indirect restorations (16,17).

### Sample preparation

The resin cement was prepared following the manufacturer's instructions and inserted into a stainless steel matrix with a cylindrical-shaped perforation measuring 4 mm in diameter and 1.2 mm in thickness (Fig. 1). A clear Mylar strip was placed over the resin cement, and an Empress 2 porcelain disc (Ivoclar, Schaan, Liechtenstein), measuring 6 mm in diameter and 2 mm in thickness, was placed on top. This thickness of the porcelain disc was selected because, generally, it is

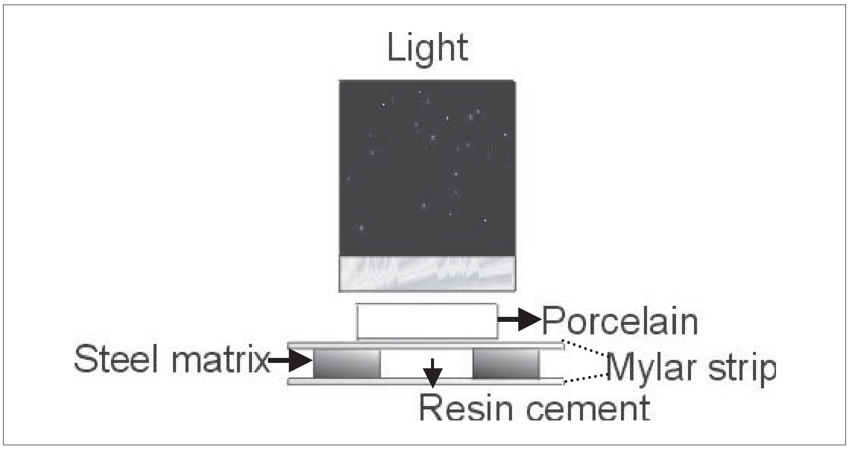


Figure 1. Diagram of the experimental set-up used for polymerization of resin cement through a porcelain disc.

Table 1. Materials used in this study, manufacturer, batch number, and shade of the materials.

| Materials and Light curing units | Manufacturer | Batch no. | Shade |
|---|---|---|---|
| Enforce – catalytic | Dentsply, Rio de Janeiro, Brazil | 67966 | - |
| Enforce – base | Dentsply, Rio de Janeiro, Brazil | 68390 | B1 |
| Empress 2 porcelain | Ivoclar, Schaan, Liechtenstein | 23878 | B3 |
| Ultraled (130 mW/cm$^2$) | Dabi-Atlante, Ribeirão Preto, SP, Brazil | 1051-5 | - |
| XL 2500 (760 mW/cm$^2$) | 3M, Sumaré, SP, Brazil | 3000165 | - |

Table 2. Vickers hardness numbers and standard deviations (VHN) of Enforce dual-cured cement.

| | LED light | | | | | | QTH light (conventional) | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Group | 1A - 60s | | 1B - 80s | | 1C -120s | | 2 A - 60s | | 2 B - 80s | | 2C - 120s | |
| | Top | Bottom | T | B | T | B | T | B | T | B | T | B |
| **Experimental*** | 43,1 (1,6) | 28,6 (3,8) | 43,0 (3,9) | 33,1 (2,4) | 44,3 (1,4) | 37,0 (2,7) | 48,0 (6,8) | 41,2 (3,2) | 46,9 (1,1) | 42,7 (4,9) | 51,1 (2,8) | 45,8 (3,1) |
| Group | 3A - 60s | | 3B - 80s | | 3C -120s | | 4 A - 60s | | 4 B - 80s | | 4C - 120s | |
| | Top | Bottom | T | B | T | B | T | B | T | B | T | B |
| **Control**** | 48,8 (1,2) | 42,5 (6,0) | 46,8 (3,8) | 45,2 (2,5) | 48,5 (4,0) | 46,1 (4,0) | 55,7 (3,9) | 46,7 (4,5) | 54,3 (1,6) | 46,9 (2,2) | 52,4 (4,0) | 49,7 (3,5) |

*Experimental: photo-activation with ceramic interposition

**Control: photo-activation with no ceramic interposition

compatible with indirect porcelain restorations such as inlays, onlays, or crowns. The resin cement was photo-cured through the porcelain with a halogen lamp (XL 2500, 3M, Sumaré, SP, Brazil) and a LED unit (Ultraled, Ribeirão Preto, SP, Brazil) at exposure times of 60, 80, and 120s. The halogen and LED power densities were 760 mW/cm$^2$ and 130 mW/cm$^2$, respectively. These power measurements were obtained using a calibrated power meter (Field Master, Coherent, USA). The same procedures were performed on the control groups (G3A, G3B, G3C, G4A, G4B and G4C), however, without interference of the porcelain disc and close to the cement surface. Each group contained five samples. The samples were then stored in distilled water in a light-proof container at 37°C for 24 hours and hardness was checked using a Vickers indentor.

### Hardness evaluation

The microhardness of the samples was obtained using the mhp 160 Microhardness Tester (Carl Zeiss, JENA) with a marker for Vickers units. The hardness value (VHN) was obtained on the top (surface near the light source) and on the bottom surface (surface distant from the light source). One indentation was made in the central part of each sample on both sides and two readings were taken on each indentation. The indentations were made using a load of 30 g for 30 s. The values were then averaged for the five samples to obtain a mean top surface value and a mean bottom surface value.

Data were submitted to four-way ANOVA and Tukey multiple comparison tests.

## Results

The mean hardness values for the top and bottom surfaces at different exposure times and photo-curing sources are shown in Table 2. Four-way ANOVA identified interactions among presence of porcelain, light source and location (top or bottom) (F=9.25 and p=0.0030). It also identified reactions between exposure time and location (F=4.45 and p=0.01). Multiple comparisons were then performed using a Tukey test at a = 0.05.

The lowest hardness values were obtained on the bottom surface when the cement was cured through the porcelain with the LED at both 60 s and 80 s. Increasing the exposure time of the LED lamp to 120 s did not yield significant differences between bottom and top surfaces (p=0.21). Differences between top and bottom surfaces were not different for the other groups, except for the control HL group when exposed for 60 s (p=0.030). That difference was caused by the increased hardness of the top surface, since the hardness value of the bottom surface at this exposure time was similar to the values obtained with the other exposure times in the same.

In control groups (photo-curing of resin cement without a porcelain disc), the hardness produced by the LED unit was statistically similar to that of the halogen lamp (p>0.05). There were no significant differences between the three exposure times for each light source on each surface (p>0.05).

In experimental groups, for the top surfaces, the hardness produced by the LED in group's 1A (60 s), 1B (80 s), and 1C (120 s) did not show statistically significant differences among them (p>0.05). In group 2 (HL) the three exposure times (60 s, 80 s, and 120 s) produced similar hardness (p>0.05) values. Although the hardness values produced by the LED unit were slightly inferior to those of the Halogen LCU groups, there were no significant differences between them (p>0.05).

For bottom surfaces, the lowest hardness value was obtained when the cement was photo-cured through the porcelain with the LED at 60 s. Increasing the exposure time of the LED lamp to 80 s resulted in differences with the halogen lamp at 60 s (p=0.09) that were no longer significant; however, it there were significant differences with the halogen lamp at both 80 s (p=0.01) and 120 s (p=0.0002). Otherwise, the LED at 120 s produced a hardness value significantly similar to those of the halogen lamp at both 60 s (p=0.97) and 80 s (p=0.69). For groups 1 (LED) and 2 (HL), the three exposure times also showed similar hardness values in bottom surfaces (p>0.05). Group 2C (HL, 120s) produced hardness values significantly higher than those of the LED groups (p<0.05).

## Discussion

Since 1995, LED units have been suggested for use in dentistry as an alternative for photo-curing of resin materials which contain photoinitiators with an absorption spectrum between 450-500 nm (11,14). In contrast with halogen lamps, the LED unit does not produce visible light by heating metal filaments, but uses junctions of doped semiconductors. As a result, LED units produce blue light that is focused to a small spectral range comparable with halogen LCUs. Furthermore, 100% of the energy emitted by blue LED is inside the spectrum of camphoroquinone (6,10,14,18). In contrast, almost 80% of the total energy of halogen LCU is outside the useful curing range (10). However, LED units present a lower intensity than conventional halogen lamps, which is an important clinical condition for photopolymerization of indirect restorations.

There are many factors that affect light transmission in indirect restorations such as porcelain thickness, exposure time and intensity of the light source (3,19). Thus, it is very important for the practitioner to know if the LED unit is a viable alternative for photo-curing indirect restorations. The hardness test is useful to determine depth of cure and is an important indirect way to verify light source efficiency (20,21).

In this study, ceramic with a 60% volume content of lithium disilicate crystals (IPS Empress 2) was used to simulate an important clinical procedure, where the light must pass through it to photo-cure the resin cement. This porcelain is widely used for indirect restorations, since it uses a unique layering ceramic comprised of sintered glass ceramic with fluorapatite crystals, which yields restorations similar in appearance to tooth structure and with inherent fluorescence and natural translucency (22). In the present study, the photo-activation was done through a 2-mm-thick porcelain disc, which was used to approximate the conditions of the experiment to those found in clinical practice.

Although the LED unit used in this study emitted lower irradiation (130 mW/cm$^2$) than the halogen LCU (760 mW/cm$^2$), the results obtained in control groups (photo-curing without a porcelain disc) showed that the LED unit was comparable with conventional halogen LCU. The experimental groups (top surfaces), where the resin cement was photo-cured through a porcelain disc (IPS Empress 2), showed that the LED unit at different exposure times (60, 80, and 120 s), was also comparable to the halogen LCU. These results can be explained by the narrow spectrum of light that falls closely within the absorption range of camphoroquinones that initiate the polymerization process (6,10,11,14). These findings may also be related to the high translucency of the Empress 2 porcelain, which allows the light to cross it with little interference. Edelhoff, Sorensen (23) evaluated the light transmission coefficient of some ceramics and found that Empress 2 showed a high transmission coefficient (77.3%), especially when compared with In-Ceram porcelain (32.4%).

The discussion focused on the thickness of resin cement generally found in the margins of indirect restorations, 50 – 100 mm (24), because those results corresponded to the top surface of the samples. However, in most indirect systems the manufacturing process leaves a uniform space between the restoration and the teeth above the marginal area, leading to a better adjustment and producing space for the resin cement. Thus, in bottom surfaces (1.2 mm), which can present this condition, a 60 s exposure time led to a surface hardness that was significantly lower than that obtained for the top surface. This indicates that polymerization was not uniform along the 1.2 mm thick cement layer, as compared to halogen LCU groups. The results indicated that only when the exposure time of LED through the porcelain was increased to 120 s, was the hardness of the bottom surface similar to that at the top. This indicates that the ability of LED lamps to uniformly cure across a 1.2 mm thick cement layer is affected by the interposition of the porcelain. Although the 1.2 mm thick cement layer may be too different from what actually occurs in clinical situations, the results suggest that exposure time with LED lamps should be extended whenever the clinician feels that the cement layer is too thick underneath the restorations. Additionally, as discussed above, other porcelain systems present higher opacity, which could also interfere with light transmission to the cement layer. Although the LED unit emits 100% of the energy within the spectrum of the camphoroquinone (10), this finding can be explained by the reduced intensity of the light emitted by the LED unit used (130 $mW/cm^2$) and by the light attenuation in deeper areas. In deeper areas of a resin material, light attenuation results in fewer excited camphoroquinone molecules, and the probability of a collision with amine (co-initiator) decreases dramatically (25). Nagel (26) stated that the intensity of a light within a given spectral range is a measure of the number of photons emitted. An isolated photon emitted at 470 nm by a light source with a reduced intensity of light has the same energy as another photon at 470 nm that is emitted by a light source with a high intensity. Thus, when the LED light needs to photo-cure deeper areas, the results achieved in this study demonstrated that longer exposure times are required, providing enhanced opportunity for an excited camphoroquinone molecule to collide with the amine, resulting in a free radical.

The findings of this study complement the existing literature on blue LED technology and, therefore, it has great potential for use in clinical applications, especially if the new versions have a greater light intensity. However, it is important to note that the results of this work are valid for the conditions (porcelain type, resin cement, and curing units) used in the study.

Therefore, the LED unit was found to be comparable with the halogen LCU. However, in a thick resin cement layer (1.2 mm) under an Empress 2 restoration, the exposure time using the LED unit should be increased to reach acceptable hardness values compared to the halogen LCU (60 s). Thus, the LED unit is a possible alternative for photopolymerization of indirect restorations.

**Acknowledgements**

We would like to thank Dr Vanderley S. Bagnato and Marcello R. B. Andreeta of the São Carlos Institute of Physics and Dr Ricardo M. Carvalho for their productive collaboration. The authors are grateful to the Romanini laboratory for technical support. This study was supported by a grant from CNPq.

## Resumo

LOPES, Lawrence Gonzaga, FRANCO, Eduardo Batista, NAME-NETO, Abrão, HERRERA, Francyle S., MONDELLI, Rafael F. Lia e PEDROSA, Sérgio de Freitas. **Polimerização de um cimento resinoso *dual* através da cerâmica Empress 2: LED vs. luz halógena.** Oral Sci., mai./ago. 2005, vol.1, no.1, p.15-21.

O objetivo deste estudo foi avaliar a influência das fontes de luz LED e Halógena (LH) na efetividade de polimerização do cimento resinoso Enforce, quando fotoativado sob um disco cerâmico (IPS Empress 2). Três tempos de exposição (60, 80 and 120 s) foram também avaliados. Dois grupos experimentais, nos quais a polimerização do cimento foi feita através do disco cerâmico, e dois grupos controles, em que a polimerização do cimento foi realizada diretamente (sem a presença da cerâmica), foram divididos em três subgrupos (três tempos de exposição), tendo cinco amostras cada: G1A- LED 60s; G1B- LED 80s; G1C- LED 120s; G2A- LH 60s; G2B- LH 80s; G2C- LH 120s; e grupos controles: G3A- LED 60s; G3B- LED 80s; G3C- LED 120s; G4A- LH 60s; G4B- LH 80s e G4C- LH 120s. O cimento foi inserido em uma matriz de aço (4 mm de diâmetro e 1,2 mm de espessura). Nos grupos experimentais o disco cerâmico foi posicionado sobre a matriz. O cimento foi fotoativado através da cerâmica tanto com o LED, quanto com a LH. As amostras foram armazenadas em um recipiente a 37°C por 24 horas, sendo que em seguida a dureza Vickers foi determinada. Os dados foram submetidos à análise de variância a quarto critérios e teste de Tukey (p£ 0.05). Na superfície do topo todas as amostras polimerizadas com LED tiveram valores de dureza similares às amostras polimerizadas com a Luz Halógena. Na superfície de base existiram diferenças estatisticamente significantes entre fonte de luz e tempos de exposição, em que o grupo com LED (60s) proporcionou resultados inferiores aos dos grupos com LH. Por outro lado, o grupo de 120s com LED foi estatisticamente similar aos grupos de 60 s e 80 s com LH. Assim, a tecnologia LED é viável para fotoativação através de restaurações de Empress 2, entretanto, quando o cimento apresenta-se com uma espessura maior é necessário um aumento no tempo de exposição.

PALAVRAS-CHAVE: Cimento resinoso; teste de dureza; cerâmica dental; luz emitida por diodo.

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