Effect of Light-Curing in the Microhardness of Resin Cements of Dual Polymerization

Isabel Gomes*; Luis Pires Lopes**

*DMD, MS. Assistant Professor, Department of Prosthodontics, School of Dentistry, University of Lisbon, Portugal, **DMD, MS, PhD. Professor and Chair, Department of Prosthodontics, School of Dentistry, University of Lisbon, Portugal

(Gomes I, Lopes LP. Effect of Light-Curing in the Microhardness of Resin Cements of Dual Polymerization. Rev Port Estomatol Med Dent Cir Maxilofac 2010;51:133-138)

Key-words:
Resin cements;
Light curing units;
Photopolymerization;
Exposure-time;
Post-exposure polymerization;
Knoop hardness

Abstract: Objective: The aim of this study was to evaluate the influence of the type of the light curing unit, exposure time and absence of light-curing in the microhardness of two resin cements of dual polymerization. Materials and Methods: Disc-shaped samples were prepared for each resin cement. The samples were light-cured with Visilux 2500 or with Elipar FreeLight 2 for 20 or 40 seconds. In addition, samples that had not been light cured were prepared for each cement studied. Measurements of Knoop microhardness were made after 15 minutes and 24 hours. The average value of Knoop microhardness was determined to each group and the results were submitted to analysis of variance ANOVA and Tukey test ($\alpha = 0.05$). Results: Differences have been detected between light-cured and non light-cured samples, with these showing significantly lower results. Samples with 20 seconds exposure time and measurements undertaken 15 minutes post-exposure showed statistically significant lower values, compared to samples with 40 seconds exposure time and measurements made 24 hours post-exposure time. No statistically significant difference was found between the results obtained with the two light-curing units. Conclusions: Light-curing with second generation LED systems did not show higher results, comparatively to conventional Halogen light units. For both cements, microhardness was significantly higher when light--curing was undertaken and there was a significant increase in microhardness after light exposure. Dental practitioners should consider that a LED whit high intensity doesn't mean a short polymerization time nor a better polymerization in regard to a conventional halogen.

Palavras Chave:
Cimentos de resina;
Fotopolimerizadores;
Tempo de fotopolimerização;
Polimerização pós-fotopolimerização;
Microdureza Knoop

Resumo: Objectivos: Este estudo avalia a influência do tipo de fotopolimerizador, do tempo de exposição e da ausência de fotopolimerização na microdureza de cimentos de resina de polimerização dupla. Materiais e Métodos: Foram preparadas amostras em forma de disco a partir de dois cimentos de resina de polimerização dupla. As amostras foram fotopolimerizadas com Visilux 2500 ou com Elipar FreeLight 2 durante 20 ou 40 segundos. Adicionalmente, foram realizadas amostras dos dois cimentos que não foram fotopolimerizadas. Medições de microdureza Knoop foram realizadas após 15 segundos e 24 horas. O valor médio de microdureza Knoop foi determinado para cada grupo e os resultados foram analisados com ANOVA e teste de Tukey ($\alpha = 0,05$). Resultados: As amostras não fotopolimerizadas apresentaram resultados significativamente inferiores comparativamente a todas as amostras fotopolimerizadas. Para um tempo de exposição de 20 segundos e para medições realizadas após 15 minutos foram encontrados valores significativamente inferiores, comparativamente ao tempo de exposição de 40 segundos e às medições realizadas após 24 horas. Não foi encontrada diferença estatisticamente significativa entre os dois fotopolimerizadores em estudo. Conclusões: a fotopolimerização com LED de segunda geração não apresenta resultados superiores, comparativamente ao halogéneo convencional. Para ambos os cimentos em estudo a microdureza foi significativamente superior quando se realizou fotopolimerização, ocorrendo uma significativa polimerização química após a fotopolimerização. O clínico deve considerar que a polimerização com LED de alta intensidade não significa um menor tempo de exposição nem uma melhor polimerização comparativamente a um halogéneo convencional.

Correspondência para:
Isabel Gomes
E-mail: isabel.mgomes@gmail.com

INTRODUCTION

Cementation procedures influence the clinical success of indirect restorations. Luting cements must retain the restoration and seal the space between the tooth and the restoration in order to prevent marginal micro-infiltration[1]. Resin cements have gained greater popularity in the past few years mainly because of the increasing use of ceramic restorations. Such a fact is especially due to a decrease in fractures incidence as well as an increase of retention with adhesive cementation[2,3].

From the several resin cements currently available, cements of dual polymerization were developed in the attempt to combine the desirable properties of chemical and the light polymerized materials cements. Given the fact that these cements are quite often used in situations where the cement's film light-curing is not granted, due to the restoration's thickness or to light reduction caused by the dental structure, chemical activation should be effective to guarantee a suitable polymerization at the non photopolymerized areas. Yet, the suitable polymerization of these systems in the absence of direct exposure to light has been questioned in several studies[4,5,6]. The lower polymerization is associated with poor mechanical properties[7].

El-Mowafy and Rubo[8] compared direct light-curing with indirect light-curing, through resin composite spacers, in the hardness of cements of dual polymerization. The average values of Knoop microhardness obtained in the light-cured samples through spacers have always been lower than those obtained in the directly light-cured samples. For some cements, hardness values were reduced by 50% or more when the resin composite spacer thickness was 4 mm or greater.

Santos Jr. *et al.*[6] studied four resin cements of dual polymerization. Direct light-curing of samples was done with a conventional Halogen unit (550 mW/cm^2), a high intensity Halogen unit (1360 mW/cm^2) and a first generation LED unit (320 mW/cm^2). The light-curing was made with one out of three exposure times, 10, 30, and 40 seconds. For all the resin cements studied, the greatest hardness value was obtained with the high intensity halogen light curing unit for the three exposure times. Light-curing with LED and with conventional halogen resulted in similar hardness values, with a few exceptions.

In a similar study, Ozturk *et al.*[9] found no statistically significant differences in the light-curing of a resin cement of dual polymerization with a conventional halogen light and with a first generation LED light.

The objective of this study was to evaluate the influence of the type of the curing unit, a conventional Halogen and a second generation LED light, the influence of the exposure time and the absence of light-curing in the Knoop microhardness of two resin cements of dual polymerization. Additionally, microhardness was evaluated after 15 minutes and after 24 hours light exposure. The null hypothesis to be tested was that there was no difference in the Knoop hardness between the different conditions of polymerization and the time among measurements.

MATERIALS AND METHODS

Two resin cements of dual polymerization were studied, RelyX ARC (3M ESPE, St. Paul, Minneapolis, USA) and Illusion (Bisco, Schaumburg, Illinois, USA) (Table 1). Using a metal mold of 11 mm diameter and 1 mm thickness, 24 cement samples were prepared. Specimens were light-cured with one of the two light units studied, a conventional Halogen, Visilux 2500 (3M ESPE, St. Paul, Minneapolis, USA), and a second generation LED, Elipar FreeLight 2 (3M ESPE St. Paul, Minneapolis, USA). Intensity of the studied light units was previously measured, using a Radiometer (Demetron Research Corp., Danbury, USA). Visilux 2500 produced 650 mW/cm^2 irradiance and Elipar FreeLight 2 irradiance was registered above the 1000 mW/cm^2.

For each made sample, the metal mold was put on a glass lamella covered by a small (0.7 mm) Mylar strip (Du Pont). Resin cements were mixed according to the manufacturer's instructions and inserted into the ring, with the help of a metallic spatula. A Mylar strip was placed over the ring filled with cement and, above it, a thin glass lamella, in order to allow the excess cement to be drained and a smooth surface to be obtained. Afterwards, light-curing occurred using one of the two light units studied. The light-curing guide was placed directly on the glass lamella with an angle of 90º and an exposure time of 20 or 40 seconds was obtained (Figure 1).

Additionally, 6 samples of non light cured resin cements were prepared in a similar way and made from the two cements. An opaque weight (500 g) was applied on the superior glass lamella, remaining for 15 minutes after the mixture of the two components of the resin cements (Figure 2).

All samples were removed from the metallic ring immediately after light irradiation, for all light cured groups, and 15 minutes after polymerization had started, for non light cured groups. Removal of the samples was handled with care in order not to damage the surface.

Brand Name	Company	Color	Composition (Given by the manufacturer)	Share	Expiring Date
RelyX ARC (RARC)	3M ESPE, St. Paul, MN, USA	Transparent – A1	Bis-GMA, TEGDMA, canphoroquinone, Benzoil peroxide, 67,5 Zircónio/sílica (1,5μm diameter)	EKFH	05-2007
Illusion (ILLU)	Bisco, Schaumburg IL, USA	Clear	Bis-EMA, TEGDMA, 70% silica (0,7μm)	Base 0600006106 Catalyst 0600006777	05-2008

Table I - The tested materials.

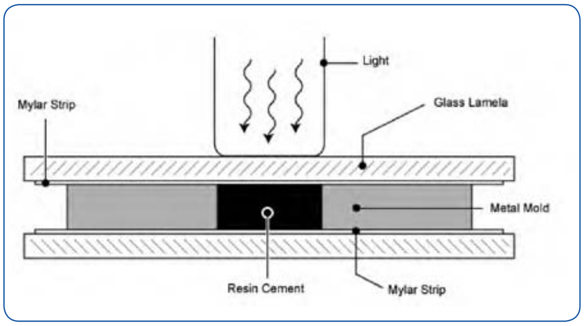

Figure 1 - Representative pattern of the preparation of photopolymerized samples.

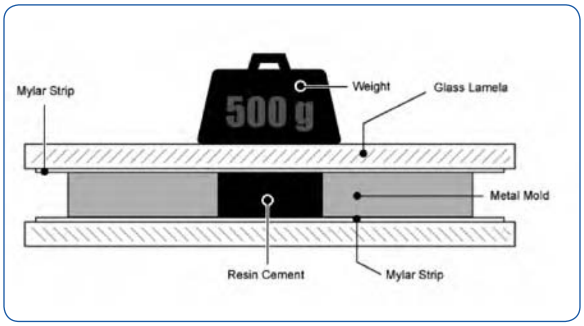

Figure 2 - Representative pattern of the preparation of non light-cured samples.

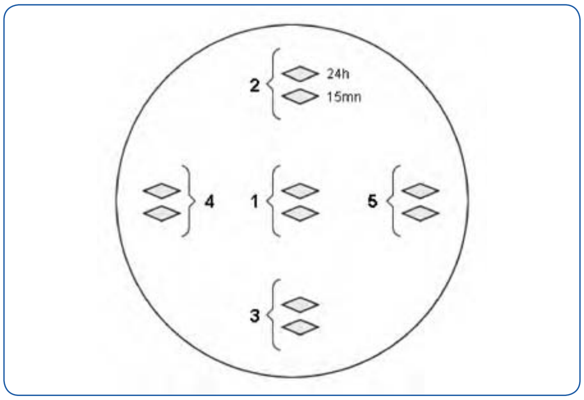

Figure 3 - Representative pattern of Knoop microhardness measurements sample made.

Five Knoop microhardness measurements were made at the surface of the samples, 15 minutes (D15) and 24 hours (D24), after the beginning of the polymerization reaction, using a Micromet 2004 hardness tester (Buehler, Lake Bluff, Illinois, USA) whit a 25 g load for 10 seconds. The five D24 measurements were followed through 0.1mm above the respective D15 measurements (Figure 3).

After light-curing and between measurements, (D15 and D24), all samples were stored in an incubator at 37º C, inside dark boxes, in a dry atmosphere.

Data of average Knoop microhardness values for D15 and D24 were obtained for each group and analyzed with a three-way analysis of variance (ANOVA) and Tukey's multiple comparisons post-hoc test for a statistical significance level of $p<0.05$, with SPSS statistical software v. 14.0 (Statistical Package for Social Science, SPSS Inc., Chicago, Ill., USA).

RESULTS

The mean Knoop hardness and the standard derivation of both studied cements, with two curing units, at different post-exposure times (D15 and D24) is shown in Figure 4. For both cements, we can verify an increase in the mean of microhardness from D15 to D24, independent from exposure time

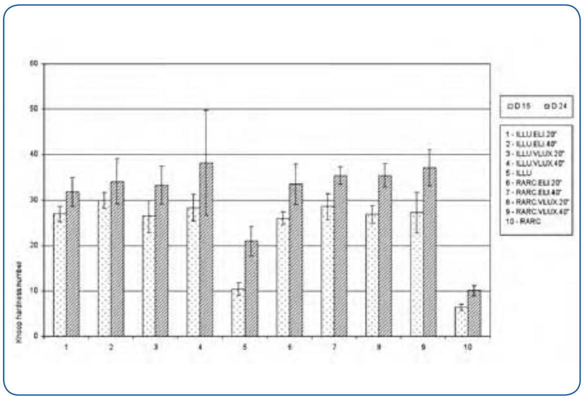

Figure 4 - Knoop microhardness: mean and standard deviations for the the different combination of resin – based cements and curing procedures.

or type of curing unit. Furthermore, this chart shows the average value of microhardness to be lower when polymerization is only chemical (group 5 and 10), comparatively to a dual-polymerization (remaining groups). Cement RelyX ARC presents lower values of microhardness when it is not light cured (group 10), on both post-exposure times (D15 and D24), comparing to non light cured cement Illusion (group 5). Twenty four hours after mixing, the mean microhardness obtained for non light-cured cement Illusion, group 5, is just 54.9% of the mean microhardness found for this cement when it is light cured for 40 seconds with Visilux 2500. Regarding RelyX ARC cement, the mean microhardness in D24 for group 10, the non light cured group is only 27.2% of the mean microhardness achieved with this cement when light cured for 40 seconds with the curing unit Visilux 2500.

For both cements, the ANOVA found statistical significant differences for the curing unit ($p < 0,001$); for exposure time ($p = 0,003$ for cement Illusion and $p = 0,005$ for RelyX ARC) and for the post-exposure time ($p < 0,001$). Additionally, a statistically significant interaction was found between curing unit and post-exposure time ($p < 0,001$) for both cements studied.

Tukey's test demonstrated statistically significant differences between groups light-cured, with Elipar FreeLight 2 or Visilux 2500, and the non light-cured groups. No statistically significant differences were found between groups light-cured with Elipar FreeLight 2 and those light-cured with Visilux 2500 for both cements.

DISCUSSION

It is important for dual-cured cements to be capable of achieving a sufficient degree of hardening with and without light-curing in order to ensure adequate polymerization of the cement in areas not accessible to the curing light[10].

The microhardness testing is the most common indirect technique used for measuring the degree of conversion of resin cements and was proved to be a reliable technique for analyzing the degree of monomer conversion[11]. However, this technique should not be used to compare the conversion extent of different resins[11] . According to such a fact, it was not an objective of this study to compare the properties of the two cements.

We would expect that, given the differences of light intensity between the two studied curing lights, higher microhardness values or equal but with a lower exposure time would be obtained with Elipar FreeLight 2. Nevertheless, the statistical analysis did not allow us to assume the expected superiority of the above mentioned LED curing light.

The light intensity measured with a radiometer was of 650 mW/cm^2 for Visilux 2500 and higher than 1000 mW/cm^2 for Elipar FreeLight 2. For an equal exposure time, a greater amount of energy was transmitted to the resin cement with Elipar FreeLight 2. Besides light intensity, the correct spectral emission is also a key factor in light-curing. In this study, no spectral characterization of light emission was made. According to the information furnished by the manufacturer, the light spectrum diffused by Elipar FreeLight 2 varies between 420 and 500 nm, with an emission peak around 460 nm, ideal to activate camphoroquinone. Even though camphoroquinone is the most frequently used photoinitiator, others might also be found, especially inside the light colors shades of composites, due to the yellow pigmentation associated to the camphoroquinone system – amine tertiary[12]. Other photoinitiators are also currently used, such as BAP (bisacilfosfine oxide) with an absorption peak of 350 nm, and PPD (1-fenil-1,2-propanodione) with a maximum absorption of 410 nm[13]. The existence of photoinitiators with absorption of lower wave-lengths will be activated by conventional halogen lights of wider specter, but not by LED lights.

A study directed by Holfmann *et al*.[14] found lower values of Vickers microhardness for two resin composites (Solitaire and Definite) when polymerized with plasma light (Apollo 93 E), compared to conventional halogen curing lights (Vivalux, Spectrum, Translux CL). The spectral radiometric output of the plasma curing light Apollo 93 E is limited to the range between 440 and 490 nm, which is optimally suited for activating camphoroquinone. However, both studied resin composites contain, in addition to camphoroquinone, photoinitiators absorbing at shorter wavelengths. These initiators can be activated by conventional halogen curing lights but not by the plasma light source.

Although the present study did not find a significant statistical difference between the two assessed curing lights, statistical analysis revealed an interaction between curing unit and post-exposure time. This interaction means that the average microhardness values were higher for curing light Elipar FreeLight 2 after 15 minutes but, after 24 hours, higher values were found for curing light Visilux 2500.

It's know that the initial low viscosity during polymerization reaction facilitates the migration of the free radicals, thus increasing the degree of cross – linking[4]. In this study the Elipar FreeLight 2 and its high intensity irradiance might have caused a rapid resin conversion, resulting in a very viscous state. Such a rapid increase in viscosity may have stopped free radicals migration, which is responsible for the polymerization reaction after light-curing, originating lower microhardness results at 24 hours when compared to a lower intensity light (Visilux 2500).

On the other hand, non light-cured samples were made to assess chemical polymerization. Ideally, resin cements of dual polymerization must be able to attain a degree of polymerization, by means of chemical reaction, similar to that obtained through light-curing. This ensures the suitable cement polymerization on the areas inaccessible to light. Even though the minimum amount of polymerization that grants success is not established, it is known that an insufficient polymerization leads to post-operative hypersensitivity, microleakage, secondary tooth caries and clinical failure due to cement's degradation in the oral cavity[15,16]. In the present study, the mean microhardness for the non light-cured groups was significantly lower than the one for the groups light-cured, which confirms previous studies[5,6].

For both cements studied, microhardness average values obtained at 24 hours were significantly higher than those obtained at 15 minutes. The same result obtained Leung, Fan and Johnston[17] which investigated post-irradiation polymerization and the effects of initial polymerization on the final polymerization of light activated resin composites. These authors concluded that the extent of polymerization was influenced by the light curing exposure time and by the post-exposure time. Microhardness increased, following light exposure, achieving a maximum value after 24 hours. Measures made after 7 days were not statistically different from those found at 24 hours.

Given this background, the dental practitioner should consider that a LED with high intensity doesn't mean a short polymerization time nor a better polymerization in regard to a conventional halogen. Moreover, all the areas of not light-cured film cement have lower microhardness and, consequently, the clinical success is not guaranteed.

CONCLUSIONS

This study investigated the effect of two curing units, a conventional halogen curing unit and a LED system, two exposure times, 20 and 40 seconds, and two post-exposures times, 15 minutes and 24 hours, on the surface microhardness of two commercial resin cements. Within the limitations of this study, it can be concluded that:

- The type of light curing units used in this study did not significantly influence the microhardness of both studied resin cements.
- Light exposure times significantly influenced the microhardness of both studied cements, being the mean values higher with 40 seconds exposure time comparatively to 20 seconds.
- Absence of light-curing resulted in significantly lower microhardness mean values, for both studied resin cements.
- After 24 hours, microhardness average values were significantly higher than those obtained after 15 minutes, for both cements.

ACKNOWLEDGEMENTS

The authors would like to thank Dr. Jorge Perdigão, Restorative Department of Minneapolis School of Dentistry, for all the lab facilities in this project, Dr. George Gomes for his valuable assistance and Dr. Ricardo Covas for his help in the data statistical analysis.

Cement materials examined in this study were kindly offered by the dental products companies 3M/ESPE and Bisco.

REFERENCES

1 - Oilo G. Sealing and retentive ability of dental luting cements. Acta Odontol Scand 1978;36:317-325.

2 - Burke FJ. The effect of variations in bonding procedure on fracture resistance of dentin-bonded all-ceramic crowns. Quintessence Int 1995;26:293-300.

3 - AL-Makramani BM, Razak AA, Abu-Hassan MI. Evaluation of load at fracture of Procera AllCeram copings using different luting cements.J Prosthodont. 2008;17:120-124.

4 - Rueggeberg FA, Caughman WF. The influence of light exposure on polymerization of dual cure resin cements. Oper Dent 1993;18:48-55.

5 - El-Mowafy OM, Rubo MH, El-Badrawy WA. Hardening of new resin cements cured through a ceramic inlay. Oper Dent 1995;24:38-44.

6 - Santos Jr GC, El-Mowafy O, Rubo JH, Santos MJ. Hardening of dual-cure resin cements and a resin composite restorative cured with QTH and LED curing units. J Can Dent Assoc 2004;70:323-328.

7 - Lovell LG, Lu H, Elliott JE, Stansbury JW, Bowman CN. The effect of cure rate on the mechanical properties of dental composites. Dent Mater 2001;17:504-511.

8 - El-Mowafy OM, Rubo MH. Influence of Composite Inlay/Onlay thickness on hardening of dual-cured resin cements. J Can Dent Assoc 2000;66:147a-d.

9 - Ozturk N, Usumez A, Usumez S, Ozturk B. Degree of conversion and surface hardness of resin cement cured with different curing units. Quintessence Int 2005;36:771-777.

10 - Vrochari AD, Eliades G, Hellwig E, Wrbas KT. Curing efficiency of four self-etching, self-adhesive resin cements. Dent Mater. 2009; 25:1104-1108.

11 - Ferracane JL. Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. Dent Mater 1985;1:11-14.

12 - Felix C, Price R, Andreou P. Effect of reduced exposure times on the microhardness of 10 resin composites cured by high – power LED and QTH curing lights. J Can Dent Assoc 2006;72:147.

13 - Stansbury JW. Curing dental resins and composites by photopolymerization. J Esthet Dent 2000;12:300-308.

14 - Hofmann N, Hugo B, Schubert K, Klaiber B. Comparison between a plasma arc light source and conventional halogen curing units regarding flexural strength, modulus, and hardness of photoactived resin composites. Clin Oral Investig 2000;4:140-147.

15 - McComb D. Adhesive luting cemets – classes, criteria and usage. Compend Contin Educ Dent 1996;17:759-773.

16 - El-Badrawy WA, El-Mowafy OM. Chemical versus dual curing of resin inlay cements. J Prosthet Dent 1999;73:515-524.

17 - Leung RL, Fan PL, Johnston WM. Post-irradiation polymerization of visible light-activated composite resin. J Dent Res 1983;62:363-365.