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Effect of Light Curing Units on Knoop Hardness of Resin-Modified Glass-Ionomer Cements

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Research Article

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ABSTRACT

Background: Different light curing units are used in the dental clinic, and they may produce different hardness values on the surface of resin-modified glass ionomer cements (RMGICs). The purpose of this study was to assess surface hardness of a nanofilled (KetacTM N100 - 3M/ESPE) and three commonly used RM GICs (VitremTM- 3M/ESPE, Vitro Fil LC[®] - DFL, Resiglass[®] - Biodinamica) using two different light curing systems: quartz tungsten halogen (QTH) and light-emitting diode (LED).

Methods: Sixteen specimens of each material were made according to manufacturer's instructions, inserted in disc-shaped molds (ø6mm; 2mm thick). The half (eight specimens) of each material were light cured using halogen light - Optilux 401, Demetron or LED- LEDemetron I, Kerr) for 20 seconds, with exception of Vitremer (40 seconds). The specimens were stored in artificial saliva for 24h, at 37°C. The surfaces were wet polished. Five Knoop Hardness Number (KHN) measurements were taken from on the upper surface with 25 g load and 30s dwell time with a Knoop indentator each sample and 7 days after.

Results: The means of KHN after 7 days were (QTH; LED), respectively: Vitremer (44.12; 40.68); N100 (35.14; 29.02); Vitro Fill LC (29.10; 30.71); Resiglass (37.90; 26.80). Vitremer showed significantly higher KHN values. No significant difference was found between the KHN in relation to light unit.

Conclusions: The type of light source has no influence on surface hardness of RM GICs.

INTRODUCTION

A greater demand for esthetic dentistry and the use of light-cured materials have improved general restorative treatments, thus the use of a light curing units are nowadays indispensable in dental practice. Visible light curing units are commonly used to polymerize light-sensitive restorative materials, such as composite resins, resin-modified glass ionomers, bonding systems and some temporary restorative materials. Adequate polymerization of these materials depends on the intensity of the light source, irradiance or power density, wavelength, and duration of exposure ^[1]. If no suitable degree of polymerization is reached, it can cause deficiency of marginal adaptation, loss of bond strength between material and tooth and lower surface hardness ^[2].

Composite resins are activated by light curing units (LCUs). These units are responsible for the excitation of photosensitive substances which react with the initiator reaction agent ^[1,2]. Curing technology was regularly subjected to changes during the last decades. Nowadays, four main light curing sources are available: halogen bulbs, plasma arc, argon ion lasers and light emitting diodes (LED) ^[3]. The LCU conventional quartz tungsten with halogen bulbs is (QTH LCU) made up of a quartz cristal, which involves a tungsten filament and an inert gas ^[4]. This produces a light by the incandescence of the filament. These units have filters which allow only blue light through, thus lessening the temperature of the light ^[5]. Camphorquinone, which is the most commonly

photoinitiator present in composite resins, is activated by rays in the range of 400-500 nm [6,7]. The LED LCU converts electrical energy into light by solid semi-conductors. This produces minimum temperature increase 5,6. However, LED LCUs emit higher light intensities, around 1000 mW/cm², which may also increase the temperature, thus decreasing its main advantage [8].

The assessment of LCU application is crucial Dentistry, as composite resins are frequently used as restorative materials. Also, glass-ionomer cements have been good alternatives for the recovery and maintenance of oral health in children. Resin-modified glass-ionomer cements (RMGICs) are similar to the conventional GICs, but they benefit from the cured resin. This allows a 'command' set by applying an external light source and the addition of methacrylate groups [9,10]. The formulation of the RMGICs are basically resin monomers in the liquid, such as hydrophilic resins (21-41% of hidroxyethylmetacrylate-HEMA) and methacrylate groups; and in the powder, photoinitiators that respond to visible light. Most RMGICs undergo a dual setting reaction: initially, chemical-cure acid-base reaction between silicate glass particles and the polycarboxylic acid, later, there are polymerization of free-radicals of the methacrylate groups, polymer and Hema [11,12]. This reaction decrease the sensitivity of the material towards water during its setting. Also, there is a greater length of work time as well as a faster setting of the material. New generation GICs have been recently launched onto the market. Ketac N100TM combines the knowledge of nanotechnology, having ionomer resin nanofilled added to the material.

The surface hardness measured after the setting of the material is used to predict its wear resistance and its ability to abrade by opposing dental structures [13]. As surface hardness tests are considered a good indicator of the degree of conversion [12], it has been used in many studies [12,14,15]. Knoop hardness tests are realized by a piramid shaped diamond indentation, which is best suited for glass ionomer cement test [16].

Based on that, the purpose of this *in vitro* study was to assess the surface hardness of four resin-modified glass ionomer cements (RMGICs) cured by a LED LCU compared to QTH LCU. The null hypothesis is that hardness of restorative materials is not influenced by the curing method.

MATERIALS AND METHODS

For this study, 16 disc-shaped specimens with 6 mm diameter and 2 mm thickness of each material in test (Vitremer™-3M/ESPE, Vitro Fil LC® - DFL, Resiglass® - Biodinâmica, Ketac™ N100 - 3M/ESPE) were prepared using plastic molds in PVC for hardness measurement (n=64). The restorative materials were handled according to the manufacturers' instructions (**Table 1**).

Material Brand Name and Manufacturer	Composition	Time the exposition	Batch n
Ketac™ N100 (3M ESPE Dental Products, St Paul, MN, USA)	H ₂ O, methacrylate modified by acid polyalkenoic, HEMA, Bis-GMA, TEGDMA, fluoro-alumino-silicate glass, nano-particles and nano-agglomerate of surface modified.	20's	M3M3 2007-12
Vitro Fill LC® (DFL Indústria e Comércio Ltda, RJ, Brazil)	Powder: Silicate of Stontium-Alumínio, filler, Ativactors and iron oxide. Líquid: 2-Hidroxietyl Metacrilato (HEMA), aqueus solution of Acids polyacrílico and Tartaric acid, Benzoyl peroxide and Camphorquinone.	20's	07121510
Vitremer™ (3M ESPE Dental Products, St Paul, MN, USA)	Powder: fluoro-alumino-silicate glass, microencapsulated potassium persulphate and ascorbic acid, small quantities of pigments. Líquid: aqueus solution of acid polycarboxylic modified with groups methacrylates, copolymer funcional of methacrylate of acid polyacrílico and polyalkenoate, H ₂ O, HEMA, photoinitiator.	40's	0823600419
Resiglass R® (Biodinamica, PR, Brasil)	Powder: Fluorosilicate of calcium, Bárium, Alumínio, acid polyacrílico and fillers Inorgânicos. Líquid: Groups Dimethacrylates, H ₂ O and catalyts (camphorquinone).	20's	810/07

Table 1. The tested materials with their compositions, specifications and manufacturers.

The molds were placed on flat glass plates over a polyester strip and then filled with RMGICs inserted in a single portion with spatula #1 (S.S.White-Duflex). The material was covered with a polyester strip and gently pressed with another glass plate against the mold to extrude excess material. After removal of the glass slab, the specimens of each test material were randomly allocated into two groups (n=8), according to the light curing unit used. They were cured with one conventional quartz tungsten halogen QTH: Optilux 401, Demetron or with light emitting diodes LED: LEDemetron I, Kerr for 20 seconds with exception for Vitremer (40 seconds). **Table 2** shows the brand names of two LCUs, their specifications and manufactures.

Light Curing Unit and Manufacturer	Lamp	Wavelength (nm)	Ligth Intensity (mW/cm ²)
Optilux 401 Demetron	Quartz-tungsten-halogen	400-520	550
LEDemetron 1 Kerr Corporation, Danbury, CT, USA	Ligth-Emitting Diode	424-515	523

Table 2. The light curing units names, manufactures and specifications.

The LEDs LCUs that use battery were used in the maximum load during the polymerization of the specimens and the light intensity was evaluated by radiometer for LEDs (LED Radiometer – Kondortech) and the QTH LCU was calibrated with a radiometer (Curing Radiometer – Demetron, Research Corporation). The distance between the light source and sample was standardized by placing the light tip in close contact with the restoration surface during polymerization. Vitremer was the only material that received a finishing gloss (3M/ESPE) layer, as advised by the manufacturer. All specimens were prepared at controlled room temperature ($23 \pm 1^\circ\text{C}$).

Immediately after light-curing, the specimens were removed from the mold. The top surface was identified with an indelible mark and stored in a dark container submerged in liquid paraffin (Merck KGaA, Darmstadt, DE) for 24 hours, at 37°C . Prior to testing, the specimens were polished in a rotation machine (Aropol 2V, Arotec®, São Paulo, Brazil), in the presence of water, with 1200 grit silicon carbide paper (Buehler), for 3 minutes with 600 rotations per minute, until the excess uneven surface was removed. Microhardness measurements were then performed using a microhardness tester with a Knoop indenter (HMV-2T, Shimadzu Corporation, Kyoto, Japan) and a 25 g load was applied for 30 seconds. Five indentations were performed at random on the top surface of each specime, three in vertically and two horizontally ^[14]. From the five indentations, the mean KHN (Knoop Hardness Number) was calculated for each specimen. After wards, all specimens were stored in artificial saliva at pH 6, 37°C for 7 days and microhardness measurements were then repeated. The difference of KHN between both times was calculated as a percentage (%KHN) of the initial microhardness value.

The data was tested for normal distribution and homogeneity with the Kolmogorov – Smirnov test. For each sample, the difference in percentage ($\Delta\%KHN$) was calculated between the final and initial KHN value. The mean %KHN difference for each material was calculated according to the type of light used. The differences in $\Delta\%KHN$ between the materials, within each type of light unit, were analyzed using the Kruskal-Wallis test, whereas the differences in $\Delta\%KHN$ between the type of light unit used in each material was calculated using Mann-Whitney test. Two-way ANOVA for $\Delta\%KHN$ between material and light unit was carried out, followed by post-hoc comparisons (Tukey's post hoc test) when the significant main effects were indicated. The level of significance used was 5% ($\alpha = 0.05$). The statistical program SPSS 13.0 software for Windows (Statistical Package, for the Social Science; SPSS Inc, Chicago, IL, USA) was used for data analysis.

RESULTS

The mean value and standard deviations ($\pm\text{SD}$) of material's KHN are summarized in **Table 3**. There was no statistically significant difference between the values of hardness in relation the light source for each material.

Materials	Light Curing Unit			
	QTH		LED	
	24 hours	7 days	24 hours	7 days
Vitremer	$51.4 \pm 13.8^{\text{a*}}$	$44.1 \pm 11.3^{\text{a}}$	$52.4 \pm 15.0^{\text{a}}$	$40.6 \pm 10.6^{\text{a}}$
Ketac N100	$32.7 \pm 12.0^{\text{b}}$	$35.1 \pm 11.2^{\text{b}}$	$27.7 \pm 5.2^{\text{b}}$	$29.0 \pm 8.2^{\text{b}}$
Vitrofill LC	$27.6 \pm 12.4^{\text{b}}$	$29.1 \pm 9.5^{\text{b}}$	$32.5 \pm 8.8^{\text{b}}$	$30.7 \pm 6.1^{\text{b}}$
Resiglass	$37.1 \pm 14.4^{\text{b}}$	$37.9 \pm 13.8^{\text{b}}$	$31.2 \pm 8.7^{\text{b}}$	$26.8 \pm 5.8^{\text{b}}$

*Different superscript letters indicate statistically significant differences in the same column

Table 3. Means \pm SD of KHN for Each Material with the Different Curing Units at the two exposure times.

Vitremer presented the highest hardness in both curing methods analyzed ($p < 0.001$), followed by Resiglass QTH LCU, N100 QTH LCU and Vitrofill LC LED LCU.

The percentage difference in KHN was calculated by subtracting the KHN value at 7 days from 24 hours. **Figure 1** shows the mean percentage difference ($\pm 95\%$ Confidence Interval) between both measurements (24 hours and 7 days) of each material according to the light unit used. As the confidence interval of most materials overlap, it shows that there was no difference between the materials, or between the LCUs used. The Kruskal-Wallis test indicated no significant difference among mean percentage difference (%KHN) in relation to time of four materials in QTH LCU ($P < 0.05$) and LED LCU ($P < 0.05$). Mann-Whitney test revealed no significant influence of LCU in all materials when the difference percentage of the initial hardness values was compared ($p > 0.05$). No differences were found between 24 hours or 7 days, nor between the different LCUs used.

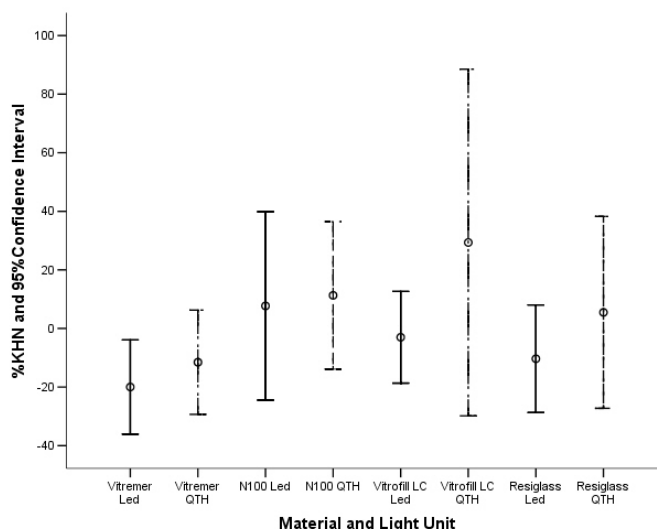


Figure 1. Mean percentage difference of hardness number (%KHN) (\pm 95% Confidence Interval) between 24 hours and 7 days measurements, according to materials and light unit.

DISCUSSION

Currently, most restorative materials depend on visible light activation of photoinitiator particles for their optimal degree of curing throughout the bulk the material. Therefore, light is an important factor on clinical success of a restoration. The shape, size, opacity, distribution and content of inorganic fillers, as well as the photoinitiator particles may significantly affect light transmission through the thickness of material increment [12]. It is well defined that mechanical properties of light-cured dental materials are dependent on the polymerization condition, light intensity, wavelength and exposure time which are critical variables for achievement of maximum curing [17].

Unfortunately, dentists have no direct means of monitoring the cure of the material surface, therefore, *in vitro* studies evaluating material surface resistance are important. By means of these studies, indirect measurements of the degree of conversion can be estimated. It has been suggested the assessment of both, top and bottom, surfaces of the materials [17], in order to assess the depth of curing in composite resins.

In this study, the specimens were 2 mm thick and only their top surface was measured to assess the curing ability of two light curing units [5]. Swift et al. [11] assessed the curing depth of five resin-modified glass ionomer restorative materials. Immediately after light activation, the upper layers of each material were harder than the deeper layers, but the degree of cure in the deeper layers improved over time. This may be a result of the chemical-curing mechanism within the materials. Also, that there was no difference between top and bottom surfaces of the specimen. For this reason, placement and curing of 2-3 mm increments remains a wise approach to the clinical use of resin-modified glass ionomer restorative materials [11,18,19].

Some studies have indicated the use of LED light curing for 2 mm thick compomers increments, in 20 seconds curing time [19,20]. A high-intensity LED LCU used for 10 seconds has resulted in relative hardness values greater than 80%, with all four restorative materials tested, indicating a sufficient degree of monomer conversion with such a short curing cycle [12].

In the present study the type of light source has no influence on surface hardness of RM GICs. Bhalla et al. [19] concluded that the curing efficacy of the LED lamp was comparable to that of conventional halogen lamp, even with a 50% reduction in cure time.

Most resin-based materials have camphorquinone photoinitiator, which is sensitive to light at the blue region of the visible spectrum [20]. Studies have proven that composite resins containing photoinitiators, which absorb light at shorter wavelengths than the LED LCUs, show lower hardness if polymerized with LED LCUs instead of halogen LCUs [21,22]. The RMGICs contains a mixture of photoinitiators, other initiators of cure that are not excited within the wavelength range covered by LED lamps. Such factors can influence the hardness of the materials. The BAPO (bis-acy1 phosphine oxide) and PPD (1-phenyl-1,2-propanodione), for example, are initiators which absorbs light at 380 nm and 410 nm, respectively. These photoinitiators may not be completely cured if LED LCUs are used [15]. In the current study, Vitro Fill LC and Resiglass R have camphorquinone in its composition. According to Fujibayashi et al. [6], camphorquinone is activated with light rays in the range of 400-500 nm. Moreover, LED LCU emit light rays in the range of 424-515 nm, which cures Vitro Fill LC.

Although no statistical significance was found between both light units, the QTH LCU seemed to have had better curing results than the LED LCU. **Table 3** shows that Vitro Fill LC had higher values when LED was used. However, the other materials had a lower average when LED LCU was used. The nanofilled RM GIC KetacTM N100 showed the lowest KHN after 7 days when LED was used (mean 29.0 KHN), but no statistical difference was found between both LCUs ($P < 0.05$). Ketac N100TM has been

launched onto the market as having nano-particles. According to the manufacturer, individual silicate charge particles (20 nm) and agglomerate of silica/zirconia (0,6 a 1,4 μm) may have advantages on the material's physical wear resistance properties, similar to hybrid composite resin.

Vitremer showed significantly higher KHN values ($P>0.05$). This may be due to the oxidation/reduction catalysts, which cause methacrylate curing after visible light exposure. Such mechanisms have been reported as "triple-cure" ^[11].

In addition, observing the values found in the current study, these materials do not comply with the specifications from the American Dental Association, which regulates the KHN for glass ionomers as at least 48.0 KHN ^[13]. When deciding which materials should be used in the clinic, the materials mechanical properties observed should also be considered. According to the manufacturer, all materials used in this study are indicated for small Class I restorations, Class II and V, sandwich technique, primary teeth restorations and provisional restorations. However, based on these results, none of the materials may be used for restorative treatments. On the other hand, hardness properties are not the only factor that should be analysed when indicating materials for restorative treatments.

One other important factor is the effect after 7 days storage in saliva artificial. The mean percentage KHN difference (%KHN) for the materials represent the full light and chemical curing ^[23]. However, none of the materials had a significant increase in microhardness, using either LCU. This finding may indicate that the hardening phase of the setting reaction still occurred after day 1, but not for all materials. As some materials had a decrease in the KHN (**Figure 1**).

RMGICs have both glass ionomer and resin components. In some RMGICs the resin monomers are separated from the polyalkenoic acid molecules, which form the basis of the ionomer. Occurs after photoinitiation of the resin components allows the materials to form a working set through the formation of a polymer skeleton. A continuing acid-base reaction establishes the hydrogel matrix of glass ionomers so the set materials contain both polymer and hydro gel elements ^[23]. This setting reaction phase occurs after the gelation phase and involves the continued formation of aluminum salt bridges ^[24].

The manufacturers should indicate on the materials package the optimum time and light wavelengths and intensity for the material's best curing results. Also, since LED devices have been launched to be used in the clinic during restorative treatments, there has been an increased interest in comparing their ability with regular QTH lamps. Future studies on physical and mechanical properties of light cured materials should be carried out with LEDs, especially on the effect of curing time on the surface hardness of the materials.

CONCLUSIONS

Vitremer showed significantly higher KHN values. The type of light source has no influence on surface hardness of RM GICs. However the manufacturers should indicate on the materials package the optimum time and light wavelength and intensity to guide the dentist to get better performance of the material used.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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