









# Assessment of influence of LED curing units used on microhardness of resin-modified glass ionomer sealants

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**Editor:** Altair A. Del Bel Cury

**Received:** June 28, 2021

**Accepted:** January 31, 2022

**Aim:** Resin modified glass ionomer (RMGI) is class of material that can be used as sealant for preventing and arresting the progression of caries in pits and fissures. As these are hybrid materials, their properties can be affected by factors related to the polymerization process. Therefore, this study aimed to evaluate the influence of different generations of LED curing units (Elipar DeepCure-L and VALO Grand) on Knoop microhardness values (KHN) of RMGI sealants (Clinpro XT and Vitremer). **Methods:** Forty cylindrical specimens (6mm  $\varnothing$  x 1 mm high) were prepared according to the manufacturer's instructions and divided into four groups (n=10) according to the type of RMGI and LED used. The KHN of the top surface of each sample was calculated 7 days after light-curing. Data were submitted to two-way ANOVA ( $\alpha = 0.05$ ). **Results:** Vitremer had higher KHN values than Clinpro XT after using both LEDs ( $p < 0.0001$ ), but especially when light-cured with the use of VALO Grand ( $p < 0.0001$ ). Whereas the KHN value of Clinpro was not influenced by the LED device ( $p > 0.05$ ). **Conclusions:** Top surface microhardness values of RGMI sealants were affected by both material composition and generations of LED curing units used. Third generation LED curing units seemed to be more efficient for the polymerization of RMGI-based sealants.

**Keywords:** Hardness. Curing lights, dental. Pit and fissure sealants.



## Introduction

Dental caries is a multifactorial disease caused by changes in the composition of bacterial biofilm, leading to an imbalance between the demineralization and remineralization processes and manifested by the formation of caries lesions in primary and permanent teeth<sup>1</sup>. Pit and fissure caries accounts for around 44% of caries in the primary posterior teeth in children and adolescents, and 90% of the caries of permanent posterior teeth<sup>1</sup>. This happens because the complex morphology of the occlusal surface makes it difficult to perform mechanical cleaning and reduces the effects of preventive measures<sup>2</sup>. Therefore, sealants that are capable of providing a physical barrier that prevents the retention of microorganism and food particles in pits and fissure have been introduced as one of many minimally invasive approaches in dentistry<sup>3,4</sup>. Their clinical efficiency has been well documented in the literature and reviews have demonstrated that they are effective for preventing both pits and fissure caries, and for minimizing the progression of non-cavitated occlusal carious lesions<sup>5,6</sup>.

According to the American Academy of Pediatric Dentistry and American Dental Association, pit and fissure sealants can be classified into two broad categories: glass ionomer (GI) sealants and resin-based sealants<sup>7</sup>. When resin is incorporated into glass ionomer, it is classified as a subcategory of material known as resin-modified glass ionomer (RMGI)<sup>1,7</sup>. The development of RMGI was proposed to improve the mechanical properties and reduce the early sensitivity to moisture of GI sealants, while preserving their clinical advantages such as esthetics, self-adhesion to dental tissue, fluoride release and thermal insulation<sup>8</sup>.

As this is a hybrid material, the setting reaction of RMGI sealant is initiated by light activation of the resin component, followed by the acid-base reaction of the ionomer component<sup>1</sup>. Although it has been suggested that the latter reaction can compensate the light attenuation that occurs in deeper areas to increase the depth of cure of RMGI<sup>9</sup>, the main mechanism responsible for the curing process of this type of material is light activation<sup>8,10</sup>. Therefore, their physical and mechanical properties can be greatly affected by factors related to the curing process<sup>11,12</sup>. Problems associated with inadequate polymerization of RMGI sealants include solubility in the oral environment, and partial or complete loss of the material resulting in recurrent caries<sup>13,14</sup>. Considering that the clinical efficiency of fissure sealants depends on their retention<sup>14,15</sup>, the relevance of the curing-process for achieving a successful outcome after sealing teeth cannot be neglected<sup>16,17</sup>.

RMGI sealants can be polymerized by using many different light sources (e.g., quartz tungsten-halogen, plasma arc, LEDs)<sup>2</sup>. However, at present LED curing units have dominated the market for many reasons since they eliminate the need for filters, weigh less and are smaller than other appliances used for light curing technologies. They also offer a more consistent radiant energy density, generate minimal heat and are long-lasting<sup>9,12,13</sup>. According to their stage of development, LED curing units can be classified into the first, second and third generations<sup>9,12</sup>.

Different methods have been used to evaluate the quality of RMGI sealant polymerization. Among them, the microhardness test has been used in many studies for

indirect assessment of RGMI polymerization and evaluation of the light sources efficiency<sup>2,8,11,18</sup>, but there is little information available about different the generations of LED curing units<sup>9</sup>. It is well known that for effective light-curing of a resin-based material, sufficient radiant exposure at the correct wavelengths of light of the photoinitiators is required<sup>12</sup>. However, the spectral radiant power derived from LED curing units varies greatly due to unique optical characteristics used within a given design<sup>16,17,19</sup>. Therefore, the influence of factors related to LED curing units on the RMGI sealants properties should also be investigated.

In view of the significant role of effective polymerization in the long-term clinical success of RGMI sealants used for caries prevention, and the recent advances in the area of light curing, the aim of this study was to evaluate the effect of different generations of LED curing units on Knoop microhardness values of RMGI. The null hypothesis tested were: (1) there would be no difference in KHN values between the RMGI sealants being evaluated; (2) there would be no difference in the KHN values of RMGI sealants cured using a second-generation LED curing unit and a third-generation LED curing unit.

## Material and Methods

Two RMGI sealants (Clinpro XT and Vitremer) and two LED curing units of different generations (Elipar DeepCure-L and VALO Grand) were used in this study. Table 1 provides details about them.

**Table 1.** Details of RGMI sealants and LED curing units used in this study

Light-curing unit	Manufacturer	Type	Emission spectrum (nm)	Irradiance (mW/cm <sup>2</sup> )
Elipar DeepCure-L	3M ESPE, St. Paul, MN, USA	2 <sup>nd</sup> generation of LED Monowave	430-480	Continuous mode: 1470 (-10%/+20%)
VALO Grand	Ultradent Products Inc, South Jordan, UT, USA	3 <sup>rd</sup> generation of LED Polywave	385-515	Standard mode: 1000 (±10%)

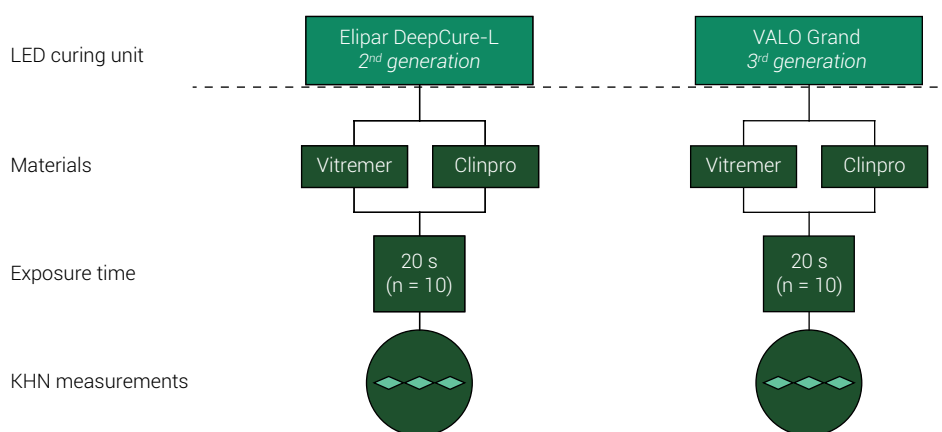
RMGI sealants	Manufacturer	Composition	Manufacturer's instructions <sup>a</sup>
Vitremer	3M ESPE, St. Paul, MN, USA	<i>Powder:</i> silane treated glass, potassium persulfate. <i>Liquid:</i> copolymer of acrylic and itaconic acids, water, HEMA, ethyl acetate and diphenyliodonium hexafluorophosphate.	Place an equal number of level powder scoops and liquid drops. Mix the powder into the liquid within 45 seconds. Light-cure for 40 s.
Clinpro XT Varnish	3M ESPE, St. Paul, MN, USA	<i>Part A:</i> silane treated glass, HEMA, water, Bis-GMA, silane treated silica, EDMAB. <i>Part B:</i> copolymer of acrylic and itaconic acids, water, HEMA, calcium glycerophosphate.	Dispense 1 click onto mixing pad. Mix for 15 seconds. Light-cure for 20s.

Notes: <sup>a</sup>In the present study, all materials were light-cured for 20 s.

Abbreviations: Monomer abbreviations: HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol A glycidyl methacrylate; EDMAB: ethyl 4-dimethyl aminobenzoate.

## Specimen preparation

Forty specimens were prepared and divided into four groups (n=10) according to material/light-curing unit combination, as shown in the experimental design (Figure 1). The RGMI sealants were manipulated according to the respective manufacturers' instructions (Table 1) and inserted into a cylindrical teflon matrix (6 mm x 1 mm thick) placed on a glass plate. After insertion, a mylar strip was placed on the surface of the unpolymerized material, and another glass plate was pressed over the strip to adapt the material completely and produce a flat surface. The tip of the LED was then placed in contact with the mylar strip on the matrix top surface, and specimens were light-cured for 20 s using one of the LED curing units according to material/light-curing unit combination (Fig.1). The radiant emittance of the LED curing units was periodically assessed using a properly calibrated radiometer (RD-7, Ecel, Ribeirão Preto, Brazil). Immediately after exposure to light, the specimens were removed from the matrix and stored in dry, lightproof receptacles until they were tested.



**Figure 1.** Experimental design of this study.

## Microhardness measurements

The microhardness test was performed 7 days after storage of the specimens, with the use of a digital Knoop hardness measuring instrument (HMV-2T E, Shimadzu Corporation, Tokyo, Japan). Three indentations were made on the top surface of all specimens: one central (defined by the location of light application) and the other two at approximately 200  $\mu\text{m}$  from the central location, under 50 kgf load for 10 s. The KHN values for each sample was recorded as the average of the three readings.

## Statistical Analysis

After descriptive and exploratory data analysis, two-way ANOVA was used to evaluate the influence of the two variables tested (RGMI sealants and LED curing units) on KHN values. The software R Core Team 2019 was used (R: A language and envi-

ronment for statistical computing. R Foundation for Statistical Computing, Vienna, Austria) at a significance level of 0.05.

## Results

As may be visualized in Table 2, the microhardness (KHN) values were significantly higher when the Vitremer ( $p < 0.0001$ ) sealer was used. For the Clinpro sealer, there was no significant difference irrespective of the LED curing unit tested ( $p > 0.05$ ). Whereas the Vitremer sealer showed higher KHN values when the VALO Grand was used ( $p < 0.0001$ ).

**Table 2.** Means (standard deviation) of microhardness (KHN) values in function of sealant and light-curing unit

LED curing units	Sealants	
	Clinpro	Vitremer
VALO Grand	35,31 (2,26) Ba	60,92 (3,86) Aa
Elipar DeepCure-L	32,30 (2,13) Ba	50,65 (1,57) Ab

Means followed by different letters (uppercase in the horizontal and lowercase in the vertical) differ from each other ( $p \leq 0.05$ ). p (sealant)  $< 0.0001$ ; p (light-curing unit)  $< 0.0001$ ; p (interaction)  $< 0.0001$ .

## Discussion

The dental light-curing unit (LCU) is an essential part of the process of light-curing a resin-based material, yet the relevance of the LCU and how it is used to achieve a successful restoration outcome is often underestimated<sup>16,17</sup>. Due to the advances in LED technology, this study aimed to evaluate the effect of different generations of LED curing units on KHN values of RGMI sealants. Only the top surface of the specimens was tested since materials used as pit and fissure sealants are applied in a thin layer on the occlusal surface<sup>3</sup>.

Vitremer exhibited higher KHN values than Clinpro, irrespective of the LED curing unit used ( $p < 0.05$ ) and, therefore, the first null hypothesis was rejected. According to the manufacturers, Vitremer contains glass filler particles of a relatively large average size ( $\sim 3\mu\text{m}$ )<sup>20</sup> corresponding to 65% by weight (according to the material safety data sheet). Whereas Clinpro is considered an unfilled RMGI<sup>1</sup>. As the physical and mechanical properties of dental resin-based materials depend on the concentration and size of filler particles<sup>19,21,22</sup>, this characteristic of Vitremer in comparison with Clinpro might have contributed to the result. Moreover, Vitremer is considered a "tri-cure" restorative material, which means that its setting reaction depends on three mechanisms: (1) the acid-base reaction between the fluoroaluminosilicate glass and the polycarboxylic acid (the same reaction as in a conventional glass ionomer), (2) a light-activated free radical polymerization of methacrylate groups of the polymer and HEMA (2-hydroxyethylmethacrylate), and (3) a chemically-initiated reaction between remaining methacrylate groups of the polymer system and HEMA (Technical Profile Vitremer). The latter is possible because a potassium persulfate/ascorbic acid redox initiation system was incorporated to its composition<sup>23</sup>.

When powder and liquid are mixed, the reaction is initiated and proceeds independent of light. Higher values of microhardness<sup>24</sup> and DC<sup>13</sup> that have been relatively stable over time have been demonstrated for Vitremer in other studies and were attributed to the enhanced physical and mechanical properties due to the complementary mechanism of cure. Whereas Clinpro has only two mechanisms of cure (acid-base, light-activated).

Furthermore, Rafeek et al.<sup>25</sup> (2008) showed that the setting process of RMGI can be benefited by heat. This may result in the accelerated maturity and improved mechanical properties of RGMI sealants<sup>26,27</sup> in addition to their enhanced adhesion to the tooth tissue of the cavity walls<sup>28</sup>. The increase in temperature of a resin-based material has been attributed to light emitted by the light source, heat released in an exothermic reaction of material hardening and its rate of polymerization<sup>16,29</sup>. Considering that Vitremer has three mechanisms of cure, one may suggest that its rate of polymerization and internal heating produced during curing could also be higher than those of Clinpro, which could also contribute to its higher KHN values, irrespective of the LED curing unit used ( $p < 0.05$ ).

Another characteristic of Vitremer that might have contributed for its results is the presence of diphenyliodonium hexafluorophosphate (DPI) in its composition. DPI is an important onium salt catalyst used to improve the reactivity of dental materials<sup>30</sup>. Although it cannot absorb light in the blue wavelength range<sup>31</sup>, in the presence of the excitatory state of camphorquinone, this co-initiator is decomposed in phenyliodonium and free phenyl radicals that can improve the photopolymerization kinetics of methacrylates, especially in ternary systems<sup>32,33</sup>. It has been demonstrated that even low concentrations of DPI participate efficiently in the monomer polymerization reducing the photo-activation time required to reach higher conversion when compared to systems without the co-initiator<sup>31</sup>. The benefit of improved mechanical properties due to increased degree of conversion by the incorporation of DPI have been demonstrated for many resin-based materials<sup>30-33</sup> and agrees with the results of this current study, even though Vitremer was light-cured for half the time recommended by the manufacturer.

Since Vitremer showed higher KHN values when VALO Grand was used ( $p < 0.05$ ), but there was no significant difference irrespective of the LED curing unit ( $p > 0.05$ ) used for Clinpro, the second null hypothesis was partially rejected. The explanation may depend on the characteristic of both the LED curing units tested and composition of the materials. Shimokawa et al.<sup>34</sup> (2018) evaluated the potential effect of four different LCUs, including VALO Grand and Elipar DeepCure-S, on the curing profile of two bulk fill resin-based composites (RBCs). Both the tip diameter and the homogeneity of the light emitted from the LCUs affected their results. They found that VALO Grand produced the most homogeneous microhardness values across top and bottom surfaces of all the RBCs tested ( $p > 0.05$ ). Whereas when Elipar DeepCure-S was used, the hardness values obtained in the central, middle and outer regions across the RBC specimens differed significantly ( $p < 0.05$ ). Moreover, they demonstrated that the light distribution of VALO Grand was more homogeneous than that of Elipar DeepCure-S. It should be noted that according to manufacturers, Elipar DeepCure-S and L offers an identical technical performance (Technical Pro-

file Elipar Deep-Cure). The main differences between the two versions are the housing and how the units are charged. Therefore, the association of a wide tip (VALO Grand: 11.5mm, Elipar DeepCure-L: 10mm) and a more homogenous light distribution may explain why Vitremer showed higher values of KHN when light-cured by VALO Grand than when using Elipar DeepCure-L ( $p < 0.05$ ). These features are especially important when light-curing a RGMI sealant because the material is applied on the total extension of pit and fissures of the occlusal surface, and it is necessary to completely cure the material to ensure long-term retention.

There is concern that high-power LED curing units, such as those used in this study, could be capable of harming the pulp and oral tissues<sup>12</sup>. Nonetheless, pit and fissure sealants are applied on the occlusal surface more thinly than a resin composite in a cavity and, therefore, the pulp is protected by the overlying dentin. Furthermore, considering that the tip of the light device cannot be placed directly on top of the sealant surface due to the morphology of fissures and cusps, this type of material could benefit from the use of high-power LED curing units, since higher irradiances can compensate the distance between the material and the light tip<sup>8,19</sup>.

Third-generation of LED curing units have further advantages over other light sources and their previous generations. Time-saving procedures are an ongoing demand for restorative application, especially in pediatric dentistry, and the development of third-generation of LED curing units has resulted from recent research focused on achieving shorter curing times without adverse consequences<sup>9,12</sup>. In this study, the fact that the Vitremer showed higher KHN values when cured with VALO Grand in standard mode for only 20 s, appears to have demonstrated the benefit of using a high-power curing unit. This could have resulted from the very thin layer of sealant and low light attenuation that tended to provide high levels of light energy within the sealant<sup>8,13,18</sup>. However, care should be taken since incident irradiance has only limited ability to compensate for the reduction in polymerization time and increase in efficiency<sup>9,19</sup>. Gonulol et al.<sup>9</sup> (2016) compared the polymerization of many tooth-colored restorative materials using three different modes of VALO and Elipar S10 as controls and showed that when VALO was used in extra power mode (3200 mW/cm<sup>2</sup>) for 6 s, insufficient polymerization was achieved in all of the tested materials. According to the cited authors, this was especially noted for RMGI sealants containing fluoride particles, because they might produce light attenuation in thicker increments that could have a negative effect on monomer conversion in deep layers.

Another advantage of the third-generation LED curing units refers to their wider spectral range of light emission. In order to be effective, it is well known that sufficient spectral radiant power must fall within the spectral range, as this is required to activate the photoinitiator(s) present in the material being used<sup>16</sup>. Considering that manufacturers rarely reveal the proprietary constituents their products contain, and that alternative photoinitiators requiring activation by lower wavelength of light have been developed and introduced in resin-based materials<sup>34</sup>, the use of broad-spectrum light sources that deliver both violet and blue light, are preferable<sup>12</sup>.

Nonetheless, the characteristics of LCUs are not the only factors that affect the quality of polymerization of resin-based sealants<sup>2,22</sup>. Different compositions, as well as

differences in the refractive indices of the organic matrix and inorganic filler components of the materials influence the transmission of visible light through them<sup>3,19</sup>. In this study, between the two RGMI sealants tested, Clinpro has the less heterogeneous mixture, mainly composed of organic matrix. This could be the reason why the differences between the LED curing units tested were less evident when based on the KHN values of this material.

The continual development of LED technology and composition of materials should be borne in mind. Moreover, microhardness cannot be the final indicator for evaluating the setting of RMGI sealants. Further studies using different mechanical and physical tests applied individually or in combination are needed in order to understand the complex relationship between polymerization efficiency and the use of different generations of LED devices, and their effect on the properties of resin-based materials.

Within the limitations of this study, it could be concluded that surface microhardness of RGMI sealants was affected by both material composition and generations of LED curing units used. Third-generation of LED curing units seemed to be more effective in relation to the polymerization efficiency of RGMI sealants than their previous generation. More information about LED curing units and composition of materials should be provided by manufacturers to enable clinicians to determine proper protocols for their particular RGMI/LED curing unit combinations.

## Acknowledgments

The authors are grateful to the Department of Restorative Dentistry at FOP-UNICAMP for to provide the use of the digital Knoop hardness measuring instrument.

## Conflicts of Interest

The authors have no proprietary, financial, or other conflict of interest of any nature or kind in any product, service, and/or company that is presented in this article.

## Data Availability

Datasets related to this article will be available upon request to the corresponding author.

## Author Contribution

**Joyce Figueiredo de Lima Marques:** Investigation, Formal analysis, Writing – Review & Editing, Final approval of the version to be published. **Laura Nobre Ferraz:** Methodology, Data acquisition, Project administration, Final approval of the version to be published. **Beatriz Kelly Barros Lopes:** Investigation, Writing – Original Draft, Final approval of the version to be published. **Tamires Aparecida Borges Vasconcelos:** Investigation, Writing – Original Draft, Final approval of the version to be published. **Thiely Roberts Teixeira:** Investigation, Writing – Original Draft, Final approval of the version to be published. **Débora Alves Nunes Leite Lima:** Resources, Review of the manuscript critically for important intellectual content, Final approval of the version to be published. **Flávio Henrique Baggio Aguiar:** Conceptualization, Methodology,



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