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Effect of light-curing time on microhardness of a restorative bulk-fill resin composite to lute CAD-CAM resin composite endocrowns

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ABSTRACT: Purpose: To evaluate the minimal irradiation time to reach a sufficient polymerization of a photopolymerizable restorative bulk-fill resin composite to lute endocrowns. **Methods:** A photopolymerizable restorative bulk-fill resin composite (Filtek One Bulk Fill) was submitted to direct light-curing by a high power LED light-curing unit for 20 seconds as the positive control group ($n = 10$). Five more test groups ($n = 10$) were light-cured in a natural tooth mold from three sites (buccal, palatal and occlusal) under a 9.5 mm thick nanohybrid resin composite CAD-CAM endocrown (Lava Ultimate A2 LT), for different irradiation times: 90 seconds per site, 40 seconds per site, 30 seconds per site, 20 seconds per site and 10 seconds per site. Vickers microhardness measurements were made at two different depths and test/control ratios were calculated. Ratios of 0.8 were considered as an adequate level of curing. **Results:** Analysis shows that 30 seconds \times 3 was the minimal irradiation time that presented a test/control ratio above 0.8. Quantile regressions showed that the required irradiation time to reach a test/control ratio of 0.8 at a confidence level of 95% was 38 seconds and 37 seconds for 200 μ m and 500 μ m, respectively. There was no statistically significant difference between microhardness of the two depths except for the irradiation time of 10 seconds. A 120-second (40 seconds per buccal, palatal and occlusal site) light-curing of photopolymerizable bulk-fill resin composite to lute a resin composite CAD-CAM endocrown restoration can be considered sufficient to reach adequate polymerization. (*Am J Dent* 2020;33:331-336).

CLINICAL SIGNIFICANCE: Endocrowns are becoming more common as an alternative to conventional post and core crowns. Luting endocrowns with restorative photopolymerizable resin composite, instead of dual-cured resin composite cements presents multiple practical and biomechanical advantages. However, the minimal light-curing duration has not yet been established.

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Introduction

Adhesive restorations have played an important role in decreasing unnecessary removal of sound dental tissues. Such restorations can be produced through direct or indirect techniques. The indication for each technique depends on multiple factors, such as available time, extent of defect, financial considerations and required esthetics. Restoring large cavities with direct methacrylate-based composites involves polymerization shrinkage of the material and stress over the adhesive interface and the surrounding tissues, especially if proper layering is not performed.^{1,2} In the case of indirect restorations, shrinkage is confined to a thin layer of luting agent, and stress values on the remaining teeth structures are thus reduced. Another advantage for the indirect technique is an easier control of morphology since the shaping is done extra-orally. For adhesive luting of indirect restorations, three main classes of resin luting agents may be used: autopolymerizing (self-curing), photopolymerizing (light-curing) and dual-polymerizing (dual-curing). Autopolymerizing luting agents depend only on mixing of two pastes, while photopolymerizing luting agents require a light-curing source to initiate the chemical reaction. Dual-polymerizing luting agents, which are currently the most used, contain both autopolymerizing and photopolymerizing initiators. This combination is believed to guarantee an optimal polymerization of resin cements where light penetration is reduced, such as under thick indirect restorations that absorb part of the light energy.² Some studies^{3,4} showed that despite the presence of autopolymerizing initiators in such dual-

polymerizing resin cements, it is still possible to increase the conversion rate with the extra light-curing step in order to reach the most optimal material quality. Insufficient polymerization of resin cements could influence mechanical properties, bond strength, biological integration and overall prognosis of the restored tooth.² Using photopolymerizing restorative resin composites for luting indirect restorations is becoming increasingly popular due to multiple advantages.⁵ Higher viscosity of restorative resin composites along with the exclusive photopolymerizing trait facilitate the control and removal of excess material leading to a better integration of the restoration.⁶ Being highly filled materials, they also present better biomechanical properties,⁷ and higher color stability⁸ than low viscosity luting resin based cements. Some studies also showed higher bond strength⁵ and lower shrinkage and monomer release⁹ for the restorative resin composites in favor of resin luting cements. Reluctance of using photopolymerizing restorative composites for the adhesive luting of indirect restorations is due to different factors such as the presumed harder seating of the restoration and doubt in regard to an appropriate light-curing through thick restorations. To avoid the first complication, restorative resin composites are usually preheated in order to decrease the viscosity of the material and an ultrasonic tip is used to allow a better insertion of the restoration. It was shown that seating of indirect restorations luted with restorative resin composites was closer to the try-in session seating than restorations luted with dual-polymerizing resin cements.¹⁰ Concerning light propagation through the restorative material, an increased time

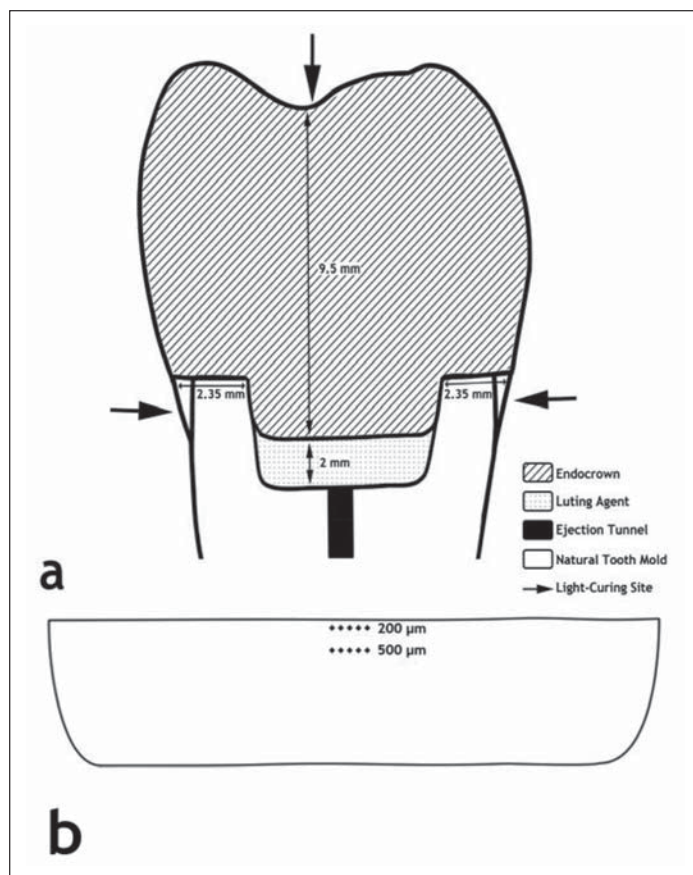


Fig. 1. Schematic representation of: **A.** bucco-palatal section of the natural tooth mold, the restoration and the different light-curing sites, **B.** mesio-distal section of a specimen after light-curing and VMH testing that shows five measurements at 200 μm and 500 μm under the occlusal surface.

of irradiation increases the delivered energy and therefore compensates light attenuation by the restoration.¹¹ Endocrowns are becoming more popular for the restoration of posterior endodontically treated teeth for their good clinical outcome and simplified conservative procedure that eliminates the need for relatively invasive post and core.^{12,13} These restorations can be considered the most extended indirect restorations since they restore a large part of the coronal portion of the tooth with an anchor in the pulp chamber, and they would therefore present a challenge for luting with a light-cured restorative resin composite. A previous study¹⁴ showed that sufficient polymerization can be achieved by using an increased irradiation time, but no information exists about the minimal time required. Conventional restorative resin composites usually have a depth of cure of 2 mm, while bulk-fill restorative materials present a chemical composition that allows a photopolymerization of 4 mm or even 5 mm in some recently launched products. Moreover, some bulk-fill materials present lower viscosity than conventional restorative resin composites which may eliminate the need for preheating the material before luting. No studies have discussed the use of bulk-fill restorative resin composites for the luting of indirect restorations. Therefore, the goal of this study was to evaluate the minimal time of light-curing required to reach sufficient polymerization of a bulk-fill resin composite placed under a large endocrown on a molar.

The first null hypothesis was that microhardness of the

luting composite would not be influenced by the light-curing duration. The second null hypothesis was that there was no difference of microhardness between 200 μm and 500 μm under the occlusal surface of the luting composite.

Materials and Methods

Similar to the study of Gregor et al,¹⁴ one natural tooth was selected as a mold in order to standardize the polymerization of the resin composite specimens through the same restoration and in a clinically relevant situation. For this purpose, a freshly extracted intact third mandibular molar was used after verifying the absence of caries and fractures. The anatomical crown was sectioned 1 mm occlusal to the CEJ and endodontic access cavity was performed to simulate a severely damaged endodontically treated tooth. A universal one component adhesive system (Scotchbond Universal^a) was used for adhesion and a restorative bulk-fill resin composite shade A2 (Filtek One Bulk Fill^a) was used to cover the pulpal floor and the axial retentions. A 4 mm deep cavity was obtained as space for a 2 mm endocrown extension and 2 mm for the luting material to be tested. In order to facilitate in later stages the removal of the luting material specimens after polymerization without altering the surfaces of the mold that will be on the path of the curing light, an ejection tunnel was created at the bottom of the cavity. A 1.5 mm cylindrical tunnel was drilled in the center going through to the furcation of the tooth that would allow the insertion of a thin instrument to push out the cured specimens. A removable plug was made using a condensation cured hard silicone (Protesil Labor^b), in order to avoid excess luting material through that tunnel.

Digital optical impression was taken with an intraoral optical scanner (Cerec Omnicam^c) and the endocrown was designed by using Cerec CAD-CAM software (SW 4.5.1). The distance between the central groove and the apical surface of the extension was 9.5 mm. Shade A2 low translucency (LT) nano resin composite CAD-CAM block (Lava Ultimate^a) was chosen to fabricate the endocrown. The shape and dimensions of the natural tooth mold and endocrown can be seen in Fig. 1. To standardize the quantity of luting material for all the specimens, the required weight to fill the 2 mm luting cavity in the natural tooth mold without excess was 0.1 g and measured for each specimen on a high precision laboratory balance (PB1502^d). For the positive control group (Group ctrl, n= 10), 0.1 g of a light-polymerizable restorative bulk-fill resin composite shade A2 (Filtek One Bulk Fill) was placed between two transparent polyester strips (Hawe Transparent Strips^e) and glass slides positioned at a 2 mm distance, then polymerized by a high power LED light-curing unit (LCU) (Valo Cordless^f) for 20 seconds to form 2 mm thick specimens. For the other experimental groups, the natural tooth mold was used and a medium thickness rubber dam (Isodam^g) was placed to simulate a clinical situation. A layer of non-oily water-soluble lubricant (Microfilm^h) was applied to the cavity and to the surface of the endocrown to facilitate removal of cured specimens, and 0.1 g of the restorative bulk-fill resin composite was placed in the mold cavity under the endocrown. It was subsequently light-cured for different durations from three sites: buccal, lingual and occlusal: Group t90 (n= 10) 90 seconds per site, Group t40 (n= 10) 40 seconds per site, Group t30 (n= 10) 30 seconds per site, Group t20 (n=

Table 1. Materials used in this study.

Material	Commercial name	Composition
Adhesive system	Scotchbond Universal	Methacryloyloxydecyl dihydrogen phosphate (MDP) monomer, dimethacrylate resins, hydroxyethyl methacrylate (HEMA), Vitrebond copolymer, filler, ethanol, water, initiators, silane
Restorative bulk-fill resin composite	Filtek One Bulk-Fill	Monomer matrix: aromatic urethane dimethacrylate (AUDMA), addition-fragmentation Monomer (AFM), urethane dimethacrylate (UDMA), dodecanediol dimethacrylate (DDDMA). Inorganic fillers: ytterbium trifluoride, silica, zirconia
Resin nano ceramic CAD-CAM block	Lava Ultimate	Silicone dioxide (SiO ₂), zirconium dioxide (ZrO ₂), reinforced resin matrix
Separating fluid	Microfilm	Glycerol, polyethylene glycol

Table 2. Mean VMH values (SD) at 200 μ m and 500 μ m under the occlusal surface. Ratio values of test mean over the positive control mean are also presented.

Curing time \times curing sites	10 s \times 3	20 s \times 3	30 s \times 3	40 s \times 3	90 s \times 3	Control (20 s direct)
200 μm						
Mean (SD)	57.8 (1.68)	57.8 (1.65)	62.8 (2.18)	66.0 (2.82)	74.9 (3.24)	76.4 (4.51)
Ratio mean 200/Positive	0.662	0.756	0.822	0.863	0.980	1.000
500 μm						
Mean (SD)	54.2*(1.31)	57.8 (1.41)	62.7 (1.88)	65.9 (3.19)	74.9 (2.16)	76.7 (2.06)
Ratio mean 500/Positive	0.706	0.753	0.817	0.859	0.976	1.000

* Statistically significant difference between the compared depths ($P < 0.001$). s = seconds.

10) 20 seconds per site, Group t10 (n = 10) 10 seconds per site. Irradiance of the LCU used in this study was measured before starting each group with a spectrometer (MARC System^b) and the mean value was 1,415 mW/cm². The diameter of the curing tip was 10 mm and the total energy delivered in 20 seconds at 0 mm distance from the spectrometer window was 28 J/cm². The specimens were then extracted from the mold, the occlusal side was marked with a permanent marker and they were immediately stored in a dark dry incubator at 37°C for 1 week. The specimens were then embedded in slow-curing transparent epoxy (EpoFixⁱ) and after setting in the dark they were sectioned in half perpendicular to the occlusal plane in the mesio-distal direction using a diamond saw (Minitomⁱ), then polished until grit 2,400 with SiC paper. Both sectioning and polishing were performed at low speed under profuse water to avoid heating. Table 1 shows the material composition.

Vickers microhardness (VMH) was measured with a microhardness testing machine (HM-124^j) at two different depths of 200 μ m and 500 μ m under the occlusal surface and using a predetermined load of 0.98 N and a dwell time of 30 seconds. Five measures were taken at each depth spaced by 100 μ m and the third measure being at the center of the specimen, which corresponds to the furthest zone from the curing light (Fig. 1). The VMH value of each specimen at a certain depth was calculated as the average of the previous five measurements.

Statistical analysis was performed with Tibco Statistica^k (version 13.3) and R statistical software.^l Normal distribution of the data was assessed by Kolmogorov-Smirnov test ($P = 0.80802$) and enabled the use of two-way ANOVA to preliminarily compare the means of each group. To study the time needed to reach at least the 80% of VMH of the directly light-cured specimen in 95% of the specimens, a quantile regression was run on the conditional fifth quantile. Quantile regression is an extension of the linear regression that does not focus on the conditional average response, but on the

conditional quantiles. The relationship between VMH values and light-curing duration is not linear so the models were estimated on the log-transformed scale; results are presented on the original data scale.

Results

The assumption of homogeneity of variance was confirmed both using Cochran's C test ($P = 0.23523$) and Levene's test ($P = 0.013$). The mean values and standard deviations of the VMH measurements at 200 μ m and 500 μ m and their corresponding test/control ratios are presented in Table 2. ANOVA showed a statistically significant difference between different light-curing durations ($P < 0.001$), but no significant difference between the two depths was found ($P > 0.05$). Fisher's least significant difference (LSD) post-hoc test showed that there was no statistically significant difference between microhardness at 200 μ m and 500 μ m except for the light-curing duration of 10 seconds ($P < 0.001$). As can be seen in Table 2, 30 seconds \times 3 is the lowest curing duration that presents an average test/control ratio above 0.8 for both 200 μ m and 500 μ m depths (0.822 and 0.817 respectively). Quantile regression showed that the required curing durations to reach a test/control ratio of 0.8 for at least 95% of specimens are 38 seconds for 200 μ m and 37 seconds for 500 μ m (Fig. 2).

Discussion

The first null hypothesis was rejected since polymerization time had a significant effect on VMH. The results of this study confirmed that for a sufficient light-curing duration, a light-cured restorative bulk-fill resin composite used as a luting material under 9.5 mm thick indirect restoration can yield 80% of VMH value of the control group which consisted of a directly light-cured specimen for 20 seconds. Even though analysis of the means, as traditionally done in previous studies,¹⁴ revealed that 30 seconds \times 3 is enough to reach a sufficient polymerization, results of 37 seconds and 38 se-

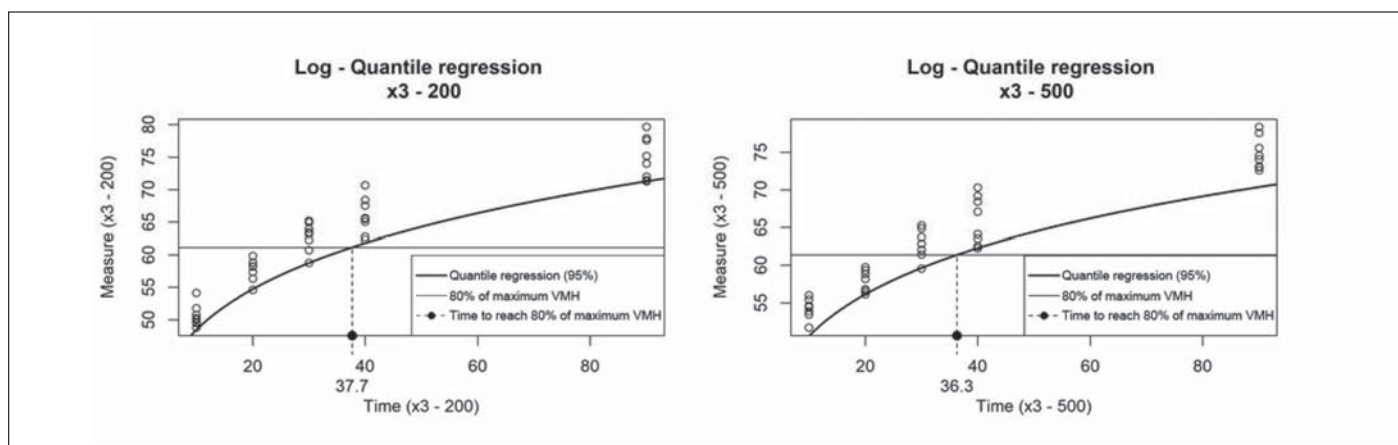


Fig. 2. VMH values at 200 μm and 500 μm under the occlusal surface, 95% quantile regression, target value and time to reach target value are also plotted.

conds per site estimated by quantile regression can be considered safer, and therefore a polymerization time of 40 seconds \times 3 can be recommended as a clinically relevant curing period. Multiple testing methods exist to evaluate the polymerization of resin composite materials. A direct method to measure the degree of conversion is by spectroscopy, such as Fourier transform infrared (FTIR) or Raman spectroscopy.^{15,16} Some indirect methods that were linked to the degree of conversion are “scrape-back” technique, which is the standard used in ISO 4049:2009, and microhardness testing, with some studies indicating that the first technique tends to overestimate the depth of cure.^{17,18} All previously mentioned techniques, including the direct measurement of degree of conversion, are not infallible indicators of the exact depth of cure. These methods fail to pinpoint the transition line between gel and glassy states of the organic matrix, along with the degree of crosslinking which is also an important indicator of the quality of cure within the composite.¹⁹ In order to obtain a detailed characterization of the quality of cure, complex tests like the use of atomic force microscopy, along with the other mentioned methods would be required. However, in the study by Leprince et al¹⁹ who used and compared these methods, they were able to show that VMH correlates very well with the degree of conversion and that it is a good clinical indicator for the properties of the composite after curing. Other studies also showed a good correlation between VMH and the degree of conversion.^{20,21} Nevertheless the limitation in that previous study regarding the VMH test was that they used the top surface of the composite as a reference, which is richer in resin and known to be less hard than the sub-surface region, and is therefore not an optimal control reference for the maximum VMH value. Polymerization is commonly considered sufficient when 90% of the maximum polymerization possible is reached, which corresponds to 80% microhardness value of the tested surface over the maximum microhardness value possible for the microhardness testing.^{18,22} Taking into consideration all previous factors, VMH was used as the testing method in the present study, and the ratio was based on the test specimen's VMH compared to the sub-surface VMH of the control group.

It is recommended by the manufacturer of the restorative bulk-fill resin composite used in this study to light-cure layers of less than 3 mm thickness for 10 seconds with a high intensity LCU ($> 1,000 \text{ mW/cm}^2$).²³ The chosen polymerization time of

20 seconds to directly polymerize 2 mm layers of the positive control group with LCU mean irradiance of $1,415 \text{ mW/cm}^2$ may thus be considered sufficient to reach the maximum microhardness of the material needed for the test/control ratio. In the present study, microhardness testing method was chosen to be able to evaluate the polymerization at two different depths under the surface of the resin composite avoiding the resin rich surface in contact with the restoration,²⁴ and to cover the range of different luting cement thicknesses. This method also allowed for measuring VMH in the furthest point from the light-curing source and thus the least favorable zone of the specimens.

The subject of luting cement thickness has been misinterpreted in multiple studies and confused with the film thickness notion of a material²⁵ that is recommended to be between 25 μm and 50 μm for luting cements, which is the thickness of a material under a specific 150 N load between two standardized glass cylinders, or with the marginal and absolute discrepancy,²⁶ where both of them do not reflect the internal luting agent thickness. Film thickness is very different from the actual luting agent thickness, which is nearest to the internal fit or gap. Default parameters for spacer thickness on common CAD-CAM systems is set to 120 μm (CEREC SW Software version 4.6), but the thickness of the luting cement itself varies a lot²⁷ and has been reported to reach a mean of $407 \pm 176 \mu\text{m}$.²⁸ This variation depends on the milling machine type, bur size and number of axes,²⁷ luting agent,¹⁰ and also the preparation and the operator.²⁷ Due to this high variability of luting agent thickness, the choice of measurements at depths 200 μm and 500 μm was made for the present study. No significant difference was found between the two measured depths except for the curing duration of 10 seconds and the second null hypothesis was also rejected. This implies that the polymerization time results could be used for luting composite thicknesses of up to 500 μm .

In cases of thick indirect restorations, literature recommends using dual-polymerizing luting cements to compensate for the attenuation of light transmittance through the restoration material.^{2,29} Even though they are currently widely used, such cements can present multiple disadvantages in terms of working time, setting time, consistency and mechanical properties compared to light-cured restorative composites.^{7,30} These factors can directly affect the excess removal process and make it more difficult, which could lead to periodontal

irritation and secondary caries.³¹ Moreover, relying only on the autopolymerizing property of the dual-polymerizing luting cement without adequate light-curing results in an insufficient degree of conversion,^{32,33} accompanied by multiple biological and mechanical complications.^{2,34} It was shown that it is possible to use photopolymerizing resin composites under thick endocrowns, by light curing for 90 seconds per occlusal, buccal and palatal site,¹⁴ but the effect of different polymerization times was not investigated. The required energy to light-cure a resin composite is around 16 J/cm²,^{35,36} so it is possible that 90 seconds per site would be more than enough to deliver this amount of energy, from where came the idea for this study to select polymerization times of 90 seconds, 40 seconds, 30 seconds, 20 seconds and 10 seconds in the present study to optimize the time of light-curing.

Other factors affecting the polymerization of resin composite luting agents were also previously investigated, like composition and photoinitiator type,²⁵ restoration material,³ filler size,²⁶ thickness³⁷ and shade, light type and irradiance.^{11,26} A study showed that different combinations of those factors have an important impact on the effective polymerization of resin composite materials, to an extent that the degree of conversion of a specific resin composite cured with a specific light-curing unit under a specific restoration material would not be affected by the increasing thickness of the restoration material from 0 to 4 mm.²⁵ The same study estimated that maximum degree of conversion of light-cured regular restorative resin composites is reachable by light-curing for 40 seconds through a 2.7 mm thick LT A3 shade composite CAD-CAM block. In the present work, the effect of light curing duration was studied on a restorative bulk-fill resin composite that was chosen due to the increased depth of cure and its lower viscosity compared to regular restorative resin composites, which may allow a shorter light curing time to facilitate the seating of the restoration. Since the objective of this study was to evaluate the curing time effect, only one type of resin composite was used to limit the variables. The choice of shade A2 for the restoration and luting composite was made on a practical basis, since it is one of the most used shades by clinicians and to be consistent with previous studies.^{38,39} The low translucency (LT) CAD/CAM resin composite block was chosen as the worst case scenario in terms of light transmittance for that common shade. One reason that has very probably led to sufficient polymerization under the thick endocrown could be the composition of the restorative bulk-fill resin composite itself containing addition-fragmentation monomer (AFM) and the polymerization through the buccal and lingual sites that would assure the majority of the required energy. Further studies are needed in order to study the necessity of light-curing from the occlusal site, especially in cases of thick restorations, and also using multiple bulk-fill resin composites. In the present study, the thickness of remaining enamel and dentin was 2.35 mm both in buccal and palatal. Light-curing through dental tissues is commonly used for photopolymerization of adhesives by transillumination in orthodontics when it is not possible to light-cure through opaque metallic brackets.^{40,41} It was shown that light curing through 8.2 mm thickness of premolars and 5.6 mm of incisors can give clinically acceptable results.^{41,42}

Therefore, using a natural tooth cavity would reproduce clinically similar conditions of light transmittance and scattering around the edges, in addition to similar conditions of pressure and contact with air that could differ in a testing setup that uses flat restorative material slices instead.

In conclusion, within the limitations of the study, the results showed that 40-second light-curing per site of a light-cured restorative bulk-fill resin composite under a nano resin composite CAD-CAM endocrown restoration can be considered sufficient to reach 80% VMH of a directly light-cured specimen for 20 seconds. This study also showed no statistically significant difference between VMH at 200 µm and 500 µm depth of the luting restorative composite except for 10 seconds light-curing per site.

- a. 3M ESPE, St. Paul, MN, USA.
- b. Vaninni Dental Industry, Florence, Italy.
- c. Sirona, Bensheim, Germany.
- d. Mettler Toledo, Greifensee, Switzerland.
- e. Kerr, Orange, CA, USA.
- f. Ultradent Products, Provo, UT, USA.
- g. Sigma Dental, Handewitt, Germany.
- h. Bluelight Analytics, Halifax, Nova Scotia, Canada.
- i. Struers, Ballerup, Denmark.
- j. Mitutoyo, Kawasaki, Japan.
- k. TIBCO Software Inc., Palo Alto, CA, USA.
- l. R foundation for Statistical Computing, Vienna, Austria.

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