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# Effect of Ceramic Veneer Opacity and Exposure Time on the Polymerization Efficiency of Resin Cements

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### Clinical Relevance

Ceramic veneers can be used for the esthetic treatment of severely discolored teeth but must have high opacity if a satisfactory result is to be achieved. This study suggests that longer exposure to light- and dual-cured cement should be used to ensure greater polymerization efficiency.



## SUMMARY

**The objective of this study was to determine the degree of conversion (DC), hardness (H), and modulus of elasticity (E) of a dual-cured resin cement, a light-cured resin cement, and a flowable resin cured through opaque or translucent ceramic with different exposure times. RelyX ARC (dual), RelyX Veneer (light-cured), and Filtek Z350 Flow resin specimens 0.5 mm thick were cured for 40, 80, and 120 seconds through 1-mm thick translucent or opaque feldspathic ceramic disks (n=10). The specimens were stored at 37°C for 24 hours. Half of each specimen was used to test the DC and the other half to test H and E. The DC was determined in a Fourier transform infrared spectrometer in absorbance mode at peaks of 1638 $cm^{-1}$ and 1610 $cm^{-1}$. H and E were determined using nanoindentation with one loading cycle and a maximum load of 400 mN. The data were analyzed with three-way analysis of variance (ANOVA), the Games-Howell test, and the Pearson correlation test (α=0.05). Statistically significant differences were found**

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**for all three factors (material, opacity, and exposure time), as well as interaction between them. The opaque ceramic resulted in lower DC, H, and E than the translucent ceramic for an exposure time of 40 seconds. An exposure time of 120 seconds resulted in a similar DC for all materials, irrespective of the opacity of the ceramic. Materials cured for 120 seconds had higher H and E than those cured for 40 seconds. The exposure time and opacity of the ceramic exerted an influence on the DC, H, and E of the materials evaluated.**

## INTRODUCTION

The use of porcelain veneers to change the color, shape, or position of anterior teeth in conservative restorations is becoming increasingly popular among today's dental practitioners. The success of this type of veneer is, to a large extent, determined by the strength and durability of the bond between the tooth surface, the luting composite, and the porcelain veneer.[1]

The materials used for luting ceramic restorations are composed of hybrid resins with a large amount of small filler particles (from 36% to 77% by weight) and a bisphenol A glycidyl dimethacrylate (Bis-GMA) matrix.[2] Commercially available cements for luting laminate veneers are usually activated by visible light or dual-cured depending on the opacity of the ceramic. The main advantages of light-cured cements are their greater color stability and working time compared with chemically cured and dual-cured systems.[3] Use of this type of cement makes it easier to remove excess material before light curing and reduces the time needed for finishing after the restorations have been luted.[1] In addition to their ease of use, light-cured resin cements for luting ceramic veneers have the further advantage that they do not use amine as a chemical initiator, which could cause the color of the material to change over time.[4] In order to take advantage of the properties of light-cured composite resins and their greater cost-effectiveness compared with resin cements, some dental practitioners have started using flowable resin composites to lute veneers. These were developed in 1996 and have the same particle size as hybrid composites but produce a mixture with lower viscosity and improved handling properties.[5] However, to date only one study has investigated the use of flowable resins as luting agents for laminate veneers.[6] Dual-cured resin cements are the most commonly used material for luting purposes. Various studies have shown that these have superior mechanical properties, such as flexural strength, modulus of elasticity, hardness, and degree of conversion, compared with cements that are exclusively light-cured or chemically activated.[7-10] Dual-cured cements have the advantage of additional chemical curing in deeper areas where light is subject to greater attenuation.[7,8,11] However, there is evidence that the chemical component of cure is lower than the light-curing component,[12] and little is known about the ability of dual-cured cements to polymerize when they are used in various clinical scenarios.[13]

While a number of studies have shown that various factors such as the type, thickness, and color of the ceramic[14-16] as well as the type of curing light, curing mode, and light intensity[14,17,18] can affect the polymerization of resin cements, there are fewer studies in the literature into the effects of the opacity of ceramic restorations on the properties of resin cements.[19,20] Nevertheless, esthetic treatment of severely darkened teeth requires the use of more opaque ceramics, which can attenuate the curing light before it reaches the luting agent.

The aim of this study was therefore to investigate the influence of exposure time and the opacity of a feldspathic ceramic on the polymerization efficiency of a dual-cured resin cement, light-cured resin cement, and flowable composite using Fourier transform infrared (FTIR) spectroscopy and nanoindentation. The hypotheses investigated in the study were 1) that exposure time and the opacity of the ceramic would not affect the polymerization efficiency of the luting cements and 2) that the polymerization efficiency of the flowable composite would be similar to that of the dual-cured and light-cured cements.

## MATERIALS AND METHODS

In order to simulate the ceramic veneers, Noritake EX-3 feldspathic porcelain discs (Noritake Dental Supply Co Limited, Tokyo, Japan) 10 mm in diameter and 1.0 mm in thickness were made with an opaque ceramic (O) in shade OBA3 and a translucent ceramic (T) in shade BA3.

Three types of materials were used: a dual-cured resin cement (RelyX ARC, 3M-ESPE, St. Paul, MN, USA), a light-cured resin cement (RelyX Veneer, 3M-ESPE), and a flowable composite (Filtek Z350 Flow, 3M-ESPE), all in shade A3. Table 1 shows the materials with their respective manufacturers, type, composition, and filler content. The resin cements were handled in accordance with the manufacturers' instructions for luting ceramic veneers.

Table 1: *Materials Used in the Study*

| Material | Manufacturer | Type | Composition | Filler |
|---|---|---|---|---|
| RelyX ARC | 3M ESPE, St Paul, MN, USA | Dual-cured resin cement | Bis-GMA, TEGDMA, zirconia/silica filler, pigments, benzoyl peroxide, amine, and photoinitiator | 67.5 wt% |
| RelyX Veneer | 3M ESPE, St Paul, MN, USA | Light-cured resin cement | Bis-GMA, TEGDMA, zirconia/silica filler | 66.0 wt% |
| Filtek Z350 Flow | 3M ESPE, St Paul, MN, USA | Flowable composite | Bis-GMA, Bis-EMA, TEGDMA, zirconia/silica filler | 65.0 wt% |

*Abbreviations: Bis-EMA, bisphenol A ethoxylated dimethacrylate; Bis-GMA, bisphenol A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate.*

The specimens were produced using a polytetrafluoroethylene (Teflon) mold 8 mm in diameter and 0.5 mm in thickness. The mold was placed on a glass plate with black adhesive paper to reduce the amount of light reflected from the benchtop surface onto the specimens. After the material had been inserted, a Mylar strip was positioned on top of it to ensure a smooth surface. A glass slide was pressed onto the strip for 15 seconds with a pair of locking pliers to allow the material to flow out and so keep a standard specimen thickness. The glass slide was then removed and the ceramic disc positioned over the assembly. Half of the specimens were light-cured through the opaque ceramic disc and the other half through the translucent ceramic disc; the purpose of both discs was to simulate ceramic veneers. Light curing was carried out with a conventional halogen curing light (Optilux 501, Demetron Corp, Orange, CA, USA) with an irradiance of 550 mW/cm$^2$ for 40, 80, and 120 seconds. Ten specimens were prepared for each of the experimental conditions.

Immediately after light curing, the specimens were stored in lightproof containers in a high relative humidity at 37°C for 24 hours to avoid additional exposure to light. Each specimen was cut down the middle with a diamond disc to form two halves; one half was used to determine the degree of conversion, and the other was used for nanoindentation tests.

The degree of conversion was measured using a FTIR spectrometer (Spectrum 100, Perkin-Elmer Corp, Norwalk, CT, USA) with an attenuated total reflectance accessory. The spectra were recorded with 20 scans at a resolution of 2 cm$^{-1}$ in the 1500 to 1800 cm$^{-1}$ band. Monomer conversion for all the materials was calculated using the standard method for monitoring the change in the ratios of the aliphatic to aromatic C = C absorption peaks at 1636 cm$^{-1}$ and 1610 cm$^{-1}$, respectively, in the uncured and cured forms according to the following equation:

$$GC = 100 \times 1 - \frac{C = C\,1636\,C/C = C\,1610\,C}{C = C\,1636\,U/C = C\,1610\,U}$$

where $C$ is the absorption peak of the cured material and $U$ the absorption peak of the uncured material.

The data were recorded in a spreadsheet (Excel 4.0, Microsoft Corp, Redmond, WA, USA) and used to calculate the descriptive statistics.

The nanoindentation tests were carried out on the other halves of the discs with an XP nanoindenter (MTS Systems Corporation, Oakridge, OK, USA) using a matrix with nine indentations, one loading cycle, a maximum load of 400 mN, and a Berkovich tip. The hardness and moduli of elasticity of the materials were determined from the curves of the applied load vs penetration depth at the surface of the sample according to the method described by Oliver and Pharr.[21]

Statistical analysis was performed with the SPSS 15.0 statistics program (SPSS Inc, Chicago, IL, USA) using three-way ANOVA (material, opacity, and exposure time), the Tukey test, and the Pearson correlation test with a significance level of 5%.

## RESULTS

Degree of conversion (DC), hardness (H), and modulus of elasticity (E) were found to differ significantly with material, opacity of the ceramic, and curing time, and there was significant interaction between material and opacity ($p<0.05$). The Pearson correlation test revealed a strong correlation between DC and H ($r^2$=0.83) and between DC and E ($r^2$=0.82) and a very strong correlation between H and E ($r^2$=0.97).

Table 2: *Mean Values (SD) of the Degree of Conversion, Hardness, and Modulus of Elasticity of the Materials Evaluated Using Different Exposure Times*

| | Material | Ceramic | 40 s (SD) | | 80 s (SD) | | 120 s (SD) | |
|---|---|---|---|---|---|---|---|---|
| **Degree of conversion, %** | RelyX ARC | Translucent | 73.21 | (1.88) | 74.93 | (1.15) | 76.06 | (1.85) |
| | | Opaque | 71.70 | (1.42) | 73.16 | (2.18) | 76.69 | (2.85) |
| | RelyX Veneer | Translucent | 69.16 | (2.37) | 71.29 | (2.07) | 73.05 | (2.40) |
| | | Opaque | 64.39 | (2.20) | 68.04 | (1.41) | 68.99 | (1.79) |
| | Filtek Z350 Flow | Translucent | 63.91 | (1.74) | 66.70 | (1.33) | 68.25 | (1.28) |
| | | Opaque | 63.86 | (0.91) | 65.25 | (0.95) | 68.36 | (0.93) |
| **Hardness, GPa** | RelyX ARC | Translucent | 0.47 | (0.02) | 0.49 | (0.01) | 0.50 | (0.01) |
| | | Opaque | 0.43 | (0.02) | 0.48 | (0.01) | 0.51 | (0.03) |
| | RelyX Veneer | Translucent | 0.37 | (0.02) | 0.40 | (0.02) | 0.43 | (0.02) |
| | | Opaque | 0.36 | (0.01) | 0.39 | (0.01) | 0.41 | (0.01) |
| | Filtek Z350 Flow | Translucent | 0.37 | (0.01) | 0.40 | (0.01) | 0.43 | (0.01) |
| | | Opaque | 0.31 | (0.02) | 0.37 | (0.01) | 0.40 | (0.01) |
| **Modulus of elasticity, GPa** | RelyX ARC | Translucent | 9.72 | (0.22) | 10.00 | (0.15) | 10.24 | (0.18) |
| | | Opaque | 8.99 | (0.48) | 9.75 | (0.26) | 10.21 | (0.39) |
| | RelyX Veneer | Translucent | 7.86 | (0.37) | 8.20 | (0.50) | 8.61 | (0.35) |
| | | Opaque | 7.73 | (0.22) | 8.26 | (0.12) | 8.50 | (0.15) |
| | Filtek Z350 Flow | Translucent | 7.79 | (0.14) | 8.42 | (0.17) | 8.52 | (0.15) |
| | | Opaque | 6.83 | (0.35) | 7.82 | (0.16) | 8.17 | (0.15) |

Table 2 and Figures 1 to 3 show the mean and standard deviations of the degree of conversion, hardness, and modulus of elasticity for the luting agents investigated.

## Degree of Conversion

When the variable ceramic opacity alone was considered, the DC was significantly higher with the translucent ceramic ($p<0.05$). Considering only the exposure time, the DC increased significantly when this variable was increased from 40 to 120 seconds. When exposure time and ceramic opacity were considered together, an exposure time of 120 seconds resulted in a statistically higher DC irrespective of the opacity of the ceramic, and the DC was not significantly different from that obtained with an exposure time of 80 seconds and a translucent ceramic ($p>0.05$). The opacity of the ceramic did not have a statistically significant influence ($p>0.05$) on the DC of the dual resin cement or the flowable composite.

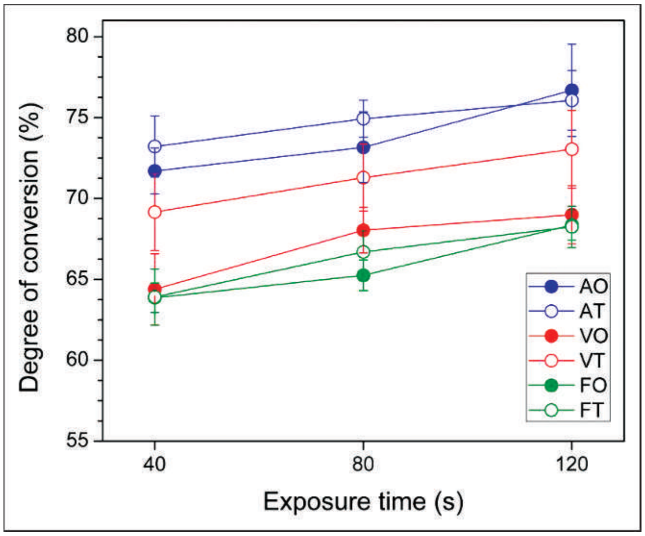


Figure 1. *Mean and standard deviations of the degree of conversion of the materials evaluated as a function of exposure time.*

When all of the variables were considered, the dual resin cement cured for 120 seconds had higher mean values of DC than all other materials, irrespective of the opacity of the ceramic ($p<0.05$). The lowest mean values of DC were observed for the flowable composite cured for less than 80 seconds through the opaque or translucent ceramic, and for the light-cured cement cured for 40 seconds through the opaque ceramic.

## Hardness

As observed for the DC, when only the opacity of the ceramic was considered, the hardness of the materials evaluated was significantly higher for the translucent ceramic and increased with increasing exposure time ($p<0.05$). When exposure time and the opacity of the ceramic were considered together, an exposure time of 120 seconds resulted in statistically higher hardness for both opacities. These mean values were not significantly different from those observed for an exposure time of 80 seconds in association with a translucent ceramic ($p>0.05$). The opaque ceramic exhibited lower hardness when an exposure time of 40 seconds was used. When the materials and the opacity of the ceramic were considered, the hardness of both light-cured materials, RelyX Veneer and Filtek Z350 Flow, was found to be affected by the opacity of the ceramic ($p<0.05$). For the variables material and exposure time, hardness increased with time, and higher mean values were observed with the dual resin cement for all exposure times evaluated.

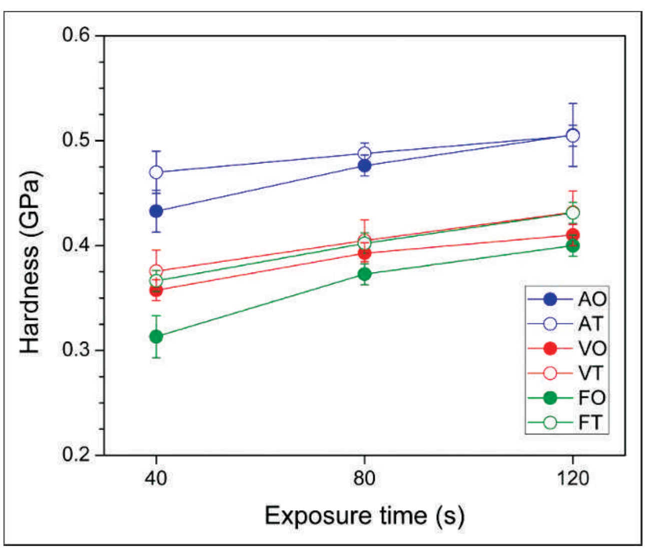


Figure 2. *Mean and standard deviations of the hardness of the materials evaluated as a function of exposure time.*

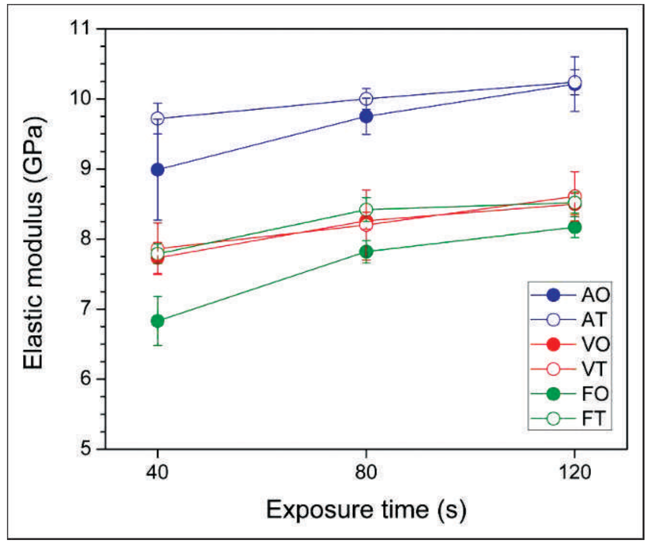


Figure 3. *Mean and standard deviations of the modulus of elasticity of the materials evaluated as a function of exposure time.*

When all variables were considered, significantly higher hardness values were achieved with the dual-cured resin cement than with the other materials, with the exception of an exposure time of 40 seconds with an opaque ceramic ($p<0.05$). The lowest mean hardness recorded was that observed for the flowable composite light-cured through opaque ceramic for 40 seconds ($p<0.05$).

## Modulus of Elasticity

There was a statistically significant increase in the modulus of elasticity both when the translucent ceramic was used and when the exposure time was increased from 40 seconds to 120 seconds ($p<0.05$). When the opacity of the ceramic and the exposure time were considered together, an exposure time of 40 seconds in association with an opaque ceramic resulted in an E value significantly lower than that for all other conditions ($p<0.05$). When the materials and the opacity of the ceramic were considered, only the E of the flowable composite Filtek Z350 Flow was found to be affected ($p<0.05$). For the variables material and exposure time, higher mean E values were observed with the dual resin cement for all exposure times evaluated, with statistically significant differences compared with the E values for the light-cured resin cement and the flowable composite for all exposure times ($p<0.05$).

As with the hardness, the highest mean E value was observed for the dual resin cement for all conditions, with the exception of the group cured for 40 seconds through opaque ceramic, which yielded similar results to those for the light-cured resin cement cured for 120 seconds through a translucent ceramic. The lowest mean E value corresponded to that observed for the flowable composite light-cured for 40 seconds using an opaque ceramic and was statistically different from the E values for all other groups evaluated in this study ($p<0.05$).

## DISCUSSION

The present study evaluated the polymerization efficiency of resin cements testing the degree of conversion, hardness, and modulus of elasticity using ceramic discs with different opacities and exposure times of light curing. The purpose in using a ceramic disc was to simulate a clinical situation, as luting agents are used underneath a restoration, leading to some attenuation of the light.[16,22-25] With this in mind, opaque and translucent feldspathic ceramic discs were used here to simulate two different clinical situations, respectively: 1) severely discolored teeth that require the use of a more opaque ceramic to mask the darkened tooth structure; and 2) teeth whose color is unaltered or only slightly altered, for which a translucent ceramic can be used.

When visible light reaches the restorative material, part of it is transmitted through the material, part is absorbed, and part is reflected at the surface. The greater the transmittance of the indirect restorative material used as a spacer, the greater the irradiance that will reach the resin cement and the greater the degree of conversion.[26] A previous study reported better polymerization in light-cured and dual-cured cements when a translucent ceramic was placed between the cement and the light source.[22] In recent studies,[27,28] less translucent ceramics were shown to result in lower hardness in resin cements than more translucent ones when the same curing mode was used. Here, the increase in the degree of conversion, hardness, and modulus of elasticity of all materials evaluated was greater when a translucent rather than an opaque ceramic was used.

The amount of light that reaches dual-cured and light-cured cements is an extremely important factor in ensuring effective polymerization. Dual-cured resin cements need to be light-cured to ensure satisfactory polymerization, although they have a chemical activator to complete the reaction.[9] Studies of the mechanical properties of resin cements cured in different modes have shown that dual-cured cements are superior to light-cured and chemically activated ones.[7,8,18,19] The dual-cured resin cement investigated in this study had better performance than the light-cured materials, particularly when exposed to light for 120 seconds.

The irradiance of the light that reaches the cement is drastically reduced when the light is transmitted across a ceramic restoration because of the effects of absorption, reflection, or transmission.[28] Longer exposure times are therefore recommended to counteract these effects.[12] The results reported here support this recommendation, as all the materials cured for 120 seconds exhibited greater polymerization efficiency than those cured for 40 seconds, a finding observed for all three properties investigated.

Incomplete polymerization of resin materials results in a low degree of conversion and large amounts of residual monomers, which can adversely affect mechanical properties and increase water sorption and solubility.[29-31] The degree of conversion of a resin material depends on factors such as the chemical structure of the monomers, the curing conditions, including light intensity and photoinitiator concentration, as well as the ambient conditions, such as atmosphere and temperature.[32] Furthermore, it has been reported that inadequate conversion as a result of low light intensity during cement curing can adversely affect the clinical performance of the restoration.[17]

Traditionally, the degree of conversion of dental materials has been determined by either direct methods, such as FTIR or Raman spectroscopy, or indirect methods, such as microhardness testing. More recently, instrumented indentation testing has become very popular in a variety of areas, primarily because it allows the mechanical behavior of materials to be characterized on a nanoscale, a process known as nanoindentation.[33] This type of testing measures the hardness and modulus of elasticity of the material directly from indentation load and displacement measurements. Another advantage of the method is that it eliminates operator-induced error as there is no need to measure the indentation area with the aid of an image.[21]

In the present study a strong correlation was observed between the degree of conversion and nanohardness of the materials evaluated. This finding is in agreement with other studies that demonstrated a positive correlation between the degree of conversion and microhardness of composite resins.[34-37] However, although both methods can be used to determine the extent of cure in resin-based materials, care should be exercised when interpreting such findings as each method is sensitive to different variables.[34] For example, the type and amount of filler particles are variables that can affect the mechanical properties of resin-based materials.[38] The materials investigated in the current study are produced by the same manufacturer and have the same type of filler particles (silica and zirconia) and very similar filler loads by weight (65.0% to 67.5%). However, the composition of the organic matrix of the flowable composite differs from that of the other materials as it contains bisphenol A ethoxylated dimethacrylate (Bis-EMA) as well as Bis-GMA and triethylene glycol dimethacrylate (TEGDMA). A recent study[39] of experimental resin cements showed that replacement of TEGDMA/Bis-GMA by various quantities of Bis-EMA does not necessarily imply significant improvements in particular mechanical properties.

The concept of energy density was adopted here since the irradiance was fixed in 550 mW/cm$^2$ with different exposure times. Other studies investigating the effects of energy density on the properties of resin cements found that the flexural strength,[40] degree of conversion,[17,40,41] and hardness[8,41-43] of dual-cured and light-cured cements were strongly dependent on this factor.

The first hypothesis in this study was rejected since both the opacity of the ceramic and exposure time affected the properties investigated. The second hypothesis was partially rejected as the flowable composite proved to be inferior to the dual-cured cement, although some of the properties of the former were similar to those of the light-cured cement. The findings indicate that flowable composites should not be used as the material of choice when cementation of an opaque ceramic veneer is indicated in a clinical setting.

Although curing times of 40 seconds are recommended by most manufacturers of luting agents, a longer exposure time should be considered to ensure sufficient conversion in cases involving more opaque ceramic restorations. In addition, dual-cured cements should be considered luting agents of choice when the esthetics are not a concern, as the presence of a chemical activator minimizes the effects of light attenuation caused by opaque ceramics.

## CONCLUSIONS

Within the limitations of this *in vitro* study, it can be concluded that:

- the opacity of the ceramic and the exposure time affected the degree of conversion, hardness, and modulus of elasticity of the luting materials evaluated;
- the dual-cured resin cement can be recommended for cementation of ceramic veneers since it demonstrated better polymerization efficiency than the light-cured cement and flowable composite; and
- when a light-cured material or an opaque ceramic are used, the minimal exposure time for polymerization should be 80 seconds.

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