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# Does ceramic translucency affect the degree of conversion of luting agents?

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## Abstract

**Objectives:** Solely light-activated luting agents have been suggested for cementing procedures with aesthetic rehabilitations, but questions remain regarding their curing potential under more opaque prosthesis. To determine the degree of carbon double bond (C=C) conversion (DC) of four categories of luting strategies when considering the interposition of lithium-disilicate ceramic laminates with different translucencies during the photo-activation procedures.

**Materials and methods:** Four different luting strategies were considered: a dual-activated resin-based cement (control, RelyX ARC, 3M ESPE), a solely light-activated resin-based cement (RelyX Veneer, 3M ESPE), a flowable resin-based composite (Filtek Z350 XT Flow, 3M ESPE), and a pre-heated (68 °C for 30 min) regular resin-based composite (Filtek Z350 XT, 3M ESPE). The DC was determined by Fourier-transformed infrared spectroscopy (n = 6), 1 min after light-activation in two conditions: (a) with direct light exposure and (b) with light exposure with the interposition of lithium-disilicate disks (e.max Press, Ivoclar Vivadent) with 1.5 mm thickness with three translucent degrees: high translucency (HT), low translucency (LT), and medium opacity (MO). The translucency parameter (TP) formula was performed to quantitatively evaluate the ceramics' translucencies using white ( $L^* = 93.7$ ,  $a^* = 1.2$ , and  $b^* = 0.8$ ) and black ( $L^* = 8.6$ ,  $a^* = -0.7$ , and  $b^* = -1.5$ ) backgrounds. The irradiance from the light curing unit (Bluephase G2, Ivoclar Vivadent) was calculated with a power meter (Ophir Optonics) with direct light exposure to the sensor and also with the interposition of the light ceramic discs. Degree of conversion data was submitted to two-way ANOVA and Tukey's test ( $\alpha = 0.05$ ).

**Results:** Translucency parameters values were 16.4, 13.4 and 12.6 for HT, LT and MO ceramics—respectively—and affected the percentage of light transmission. For all ceramic translucencies the highest DC values were observed for the dual-activated resin-based cement followed by the solely light-activated resin-based cement, the flowable composite and then by pre-heated regular composite. The ceramic's translucency influenced the DC only for the pre-heated composite.

**Conclusions:** The effect of the ceramic translucency on the curing behavior was dependent on the luting strategy. The DC was only affected for the pre-heated composite, which demonstrates lower conversion with the increased ceramic opacity.

**Keywords:** Veneers, Dental materials, Prosthodontics, Luting resins

## Introduction

Lithium-disilicate-based dental ceramic restorations are commonly used for cosmetic and oral rehabilitation treatments. Various clinical applications for crowns, conventional veneers, ultra-thin veneers, occlusal veneers, and other partial dental restorations are justified due to the range of available translucencies, higher mechanical strength compared with more traditional feldspathic glass and the ability to modify fit surfaces that allows reliable adhesion with resin-based luting materials [1–4].

Optimal polymerization of the resin-based luting material is an important goal for the clinical success of lithium-disilicate-based restorations [5]. The degree of carbon double bond (C=C) conversion (DC) is related to the mechanical properties of polymer-based materials where higher crosslink densities provide greater strength and stiffness. There are several factors of resin-based materials' composition that influence the DC as the inorganic content type, shape, and size; resin matrix type and amount; photoinitiator system type and amount. The DC determines the direct restorations' success, since lower monomer–polymer conversion corresponds to inferior mechanical properties and increased risk for leaching of toxic substances from the material [6–8]. During photoactivation of the luting agent, light is reflected at the surface and attenuated through the bulk of the ceramic material. The thickness and opacity of the lithium-disilicate ceramic is known to compromise polymerization of the resin-based luting composite [8–10]. However, studies regarding the effect of the ceramic interposition have focused mainly on traditional resin cements.

Different strategies have been proposed to bond lithium-disilicate-based restorations to the dental tissues. Usually, resin-based cements were indicated for this purpose, regardless of their activation mechanism, auto-, photo- or dual-activated materials [8, 10–12]. Previous studies have suggested that dual-cure resin-based luting composites should be avoided where cosmetics are paramount since the high content of amine-based coinitiators in their composition may induce accelerated discoloration due to oxidation processes [13–15]. Therefore, alternative strategies have been considered.

Resin-based luting composites that are polymerized solely by light energy are popular amongst dental practitioners due to wide range of color and the possible application of “try in pastes”, a water soluble—and easy to clean—glycerin-based paste that aims to reproduce the luting agent's shade. Therefore, it can be used for color matching tests and it is easy to be removed before the final luting procedure with the proper resin cement. Alternatively, the use of so-called “flowable” resin-based composites with low viscosity has also been considered [16, 17] but this approach did not become so popular among clinicians. Further, pre-heating restorative resin-based composites that lowers viscosity have already been suggested for direct restorations [18, 19] due to higher conversion and improved mechanical properties. Pre-heated resin-based composites used for lithium-disilicate luting was also encouraged as fracture resistance and longevity of the restoration was improved [20]. However, besides being attractive concerning the color stability, the consequences of solely light-activated luting materials use for low-translucent lithium-disilicate are unknown when regular restorative resin composites with regular viscosity are used with the pre-warmed technique.

## Materials and methods

### Objective

To determine the influence of lithium-disilicate translucency on the degree of carbon double-bond conversion of various resin-based luting composites. The research hypothesis was that the degree of double-bond conversion would not be dependent on the lithium-disilicate translucency only for the dual-activated resin-based cement.

### Study design

This in vitro,  $3 \times 4$  factorial study design, involved three translucency degrees of lithium-disilicate ceramic and four luting agent types.

### Lithium-disilicate ceramic discs—optical properties and light irradiance attenuation

Three lithium-disilicate ceramic disks ( $\varnothing$  10 mm, 1.5 mm thickness) with different translucencies, in A2 Shade (IPS e.max Press, Ivoclar-Vivadent, Schaan, Liechtenstein), were fabricated: high translucent (HT), medium opacity (MO) and low translucent (LT). The ceramic constituent information is provided in Table 1.

All disks were sintered according to the manufacturer's recommendations and by the same dental technician. To remove the irregularities, the surfaces finishing was carried out with polishing stone (EVE Diasync stones, Ernst Vetter GmbH) followed by graining polishing rubbers (EVE Diapol, Ernst Vetter GmbH) pink, and gray, for 5 s each. A digital caliper with 0.01 mm resolution (Digimatic Caliper 0.01–150 mm, Mitutoyo, Tokyo, Japan) was used to check each disk thickness.

In order to quantitatively determine the translucency of each ceramic disk, they were placed over standardized white ( $L^* = 93.7$ ,  $a^* = 1.2$ , and  $b^* = 0.8$ ) and black ( $L^* = 8.6$ ,  $a^* = -0.7$ , and  $b^* = -1.5$ ) backgrounds (Ceramic Colour Standard, Ceram Research Ltd., Stoke-on-Trent, Staffordshire, United Kingdom) for color measurement analysis, using an intraoral spectrophotometer (VITA Easyshade Compact, VITA Zahnfabrik, Germany) in reflectance mode and the  $L^*a^*b^*$  color space [21]. The equipment was previously calibrated in its calibration block according to the manufacturer's instructions. The probe tip ( $\varnothing$  5 mm) was placed perpendicularly, and flush, to each disk surface and the average of three measurements were recorded. In the  $L^*a^*b^*$  color space, the  $L^*$  parameter represents the lightness, where 100 is white and 0 is black, while the  $a^*$  and the  $b^*$  parameters represents the red-green and yellow-blue chromaticity coordinates respectively. Thus, the translucency parameter ( $TP$ ) formula was used [22]:

$$TP = \left[ (L_w^* - L_b^*)^2 + (a_w^* - a_b^*)^2 + (b_w^* - b_b^*)^2 \right]^{1/2}$$

where “w” refers to the  $L^*$ ,  $a^*$ , and  $b^*$  parameters values for each lithium-disilicate disk measured over the white background and “b” over the black.

The diameter of the light curing unit at (Bluephase G2, Ivoclar Vivadent, Liechtenstein) tip was measured with a digital caliper (Mitutoyo, Japan) to calculate the effective tip area in  $\text{cm}^2$ . The power (mW) was then measured using a power meter (Ophir 10A-V2-SH, Har-Hotzvim, POB 45021, Jerusalem 91450, Israel) connected to

**Table 1** Tested materials constituent information

| Luting agents  | Inorganic content fraction  | Organic matrix   | Activation system |
|--|---|--|-------------------|
| RelyX ARC  | Silane treated silica and silane treated ceramic 72 wt%   | Paste A: BisGMA, TEGDMA, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-Benzeneethanol<br>Paste B: TEGDMA, BisGMA, functionalized dimethacrylate polymer, 2-benzotriazolyl-4-methylphenol, benzoyl peroxide | Dual-activated    |
| RelyX Veneer   | Zirconia/silica and fumed silica fillers 66 wt% (0.6 mm average filler size)  | (BisGMA) and (TEGDMA) polymer  | Photo-activated   |
| Filtek Z350 XT Flow  | Zirconia/silica cluster filler and 20 nm silica filler 65 wt%   | bis-GMA, TEGDMA, and bis-EMA   | Photo-activated   |
| Filtek Z350 XT   | Non-agglomerated/non-aggregated 20 nm silica filler, non-agglomerated/non-aggregated 4 to 11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4 to 11 nm zirconia particles) average cluster particle size of 0.6 to 10 microns 78.5% by weight | bis-GMA, UDMA, TEGDMA, and bis-EMA   | Photo-activated   |
| Lithium-disilicate ceramics  |   | Crystal phase  |                   |
| IPS e.max press high translucency  | Lithium disilicate crystals Li <sub>2</sub> Si <sub>2</sub> O <sub>5</sub> (approx. 70%)  | Needle-like crystals 3 to 6 μm in length   |                   |
| IPS e.max press low translucency   |   |  |                   |
| IPS e.max press medium opacity   |   |  |                   |
| BisGMA bisphenol-A-diglycidylether dimethacrylate, TEGDMA triethylene glycol dimethacrylate, BisEMA bisphenol-polyethylene glycol dimethacrylate, UDMA urethanethyl dimethacrylate |   |  |                   |

a microprocessor (Ophir, Har-Hotzvim, POB 45021, Jerusalem 91450, Israel). Measurements were performed with the light tip juxtaposed to the power meter sensor and with the interposition of each ceramic disc. A black tape was used to surrounding area and avoid light dispersion trough any casual small gap between the light tip and the ceramic disc. Light irradiance (in  $\text{mW}/\text{cm}^2$ ) was determined by the ratio between power (in  $\text{mW}$ ) by the area ( $\text{cm}^2$ ). Additional readings were done with the Mylar strip between the light tip and the ceramic discs, but the difference was irrelevant.

### Luting agents

Four types of luting agents in A1 shade were selected for this study as shown in Table 1, a dual-activated resin-based cement (D-AC; RelyX ARC, 3M ESPE, St Paul, USA) selected as control, a photo-activated resin-based cement (P-AC; RelyX Veneer, 3M ESPE, St Paul, USA), a flowable resin-based composite (FRBC; Filtek Z350 XT Flow, 3M ESPE, St Paul, USA), and a pre-heated conventional resin-based composite (P-HRBC; Filtek Z350 XT, 3M ESPE, St Paul, USA). The conventional resin-based composite restorative

**Table 2** Lithium-disilicate discs' CIELAB parameters values over the white (w) and black (b) backgrounds, the correspondent translucency parameters (TP) and light irradiance values with the ceramic discs' interpositions

| Lithium-disilicate translucency | Optical properties |                   |                   |      | Light irradiance (Direct light irradiance = 1218 mW/cm <sup>2</sup> ) in mW/cm <sup>2</sup> (% of light maintenance) |
|---------------------------------|--------------------|-------------------|-------------------|------|--|
|                                 | L*                 | a*                | b*                | TP   |  |
| High translucency (HT)          | 94.0 <sub>w</sub>  | 0.7 <sub>w</sub>  | 14.8 <sub>w</sub> | 16.4 | 810 (66%)  |
|                                 | 78.8 <sub>b</sub>  | −0.8 <sub>b</sub> | 8.8 <sub>b</sub>  |      |  |
| Low translucency (LT)           | 89.7 <sub>w</sub>  | 1.3 <sub>w</sub>  | 22.6 <sub>w</sub> | 13.4 | 692 (57%)  |
|                                 | 79.2 <sub>b</sub>  | −0.6 <sub>b</sub> | 15.1 <sub>b</sub> |      |  |
| Medium opacity (MO)             | 83.6 <sub>w</sub>  | 4.0 <sub>w</sub>  | 38.4 <sub>w</sub> | 12.6 | 566 (46%)  |
|                                 | 76.4 <sub>b</sub>  | 0.5 <sub>b</sub>  | 28.7 <sub>b</sub> |      |  |

Values are average of three measurements performed

material was inserted in a “centrix tube” and the set (the tube already attached to the syringe, Nova DFL, Rio de Janeiro, Brazil) was placed heated in an oven to  $68 \pm 1$  °C for 30 min to reduce the viscosity [19].

#### Degree of C=C conversion

The degree of C=C conversion (DC) was determined by Fourier-transform infrared spectroscopy ( $n = 6$ ) [23], using a spectrometer equipped with an attenuated total reflectance (ATR) diamond device (Alpha, Bruker, Germany). A black circular plastic mold (0.3 mm thickness, 7 mm inner diameter) was centrally positioned over the ATR crystal and slightly overfilled with the luting materials. A Mylar strip was placed above the resin composite and flattened using the lithium-disilicate disk. After the placement of each material, an Infrared (IR) monomer spectrum was obtained using 32 scans and  $4 \text{ cm}^{-1}$  resolution. With the lithium-disilicate disk interposition, each luting material was photo-activated for 40 s. Then, the second IR spectrum was obtained 5 min after post-irradiation. The DC (%) was calculated with the following formula:

$$\% \text{ DC} = 100 \times \left\{ 1 - \left[ \frac{(C = C_{\text{cured}}/\text{aromatic}_{\text{cured}})}{(C = C_{\text{uncured}}/\text{aromatic}_{\text{uncured}})} \right] \right\}.$$

The calculation considered the differences in intensity of C=C stretching vibration (peak area) at  $1638 \text{ cm}^{-1}$  of the uncured and cured spectra. The symmetric aromatic stretching at  $1608 \text{ cm}^{-1}$  (peak area) was used as internal standard. For each luting agent, the DC was determined without and with each lithium-disilicate disk interposition.

#### Statistical analysis

The statistical analyses were conducted using SigmaPlot® 13.0 software (Systat Software). Data for the DC were analyzed by two-way analysis of variance, considering “lithium-disilicate translucency” and “resin-based luting composite” as factors. All pairwise multiple comparisons procedures were performed using the Tukey's test ( $\alpha = 0.05$ ).

**Table 3 Results of the degree of C=C conversion in percentage (% DC) depending of the different lithium-disilicate translucencies interposition during photo-activation**

| Luting composite   | DC after direct light activation | DC with light activation trough the ceramic discs |                          |                          |
|--|----------------------------------|---|--------------------------|--------------------------|
|  |                                  | High translucency (HT)                            | Low translucency (LT)    | Medium opacity (MO)      |
| Dual-activated resin-based cement (control) (RelyX ARC)        | 80.5 ± 0.9 <sup>Aa</sup>         | 82.9 ± 1.3 <sup>Aa</sup>                          | 80.3 ± 0.7 <sup>Aa</sup> | 83.9 ± 0.8 <sup>Aa</sup> |
| Photo-activated resin-based cement (RelyX Veneer)              | 73.9 ± 3.2 <sup>Ab</sup>         | 74.1 ± 2.7 <sup>Ab</sup>                          | 73.3 ± 5.7 <sup>Ab</sup> | 74.6 ± 2.0 <sup>Ab</sup> |
| Flowable resin-based composite (Filtek Z350 XT Flow)           | 68.9 ± 2.5 <sup>Ac</sup>         | 67.9 ± 1.5 <sup>Ac</sup>                          | 65.4 ± 2.3 <sup>Ac</sup> | 67.4 ± 3.8 <sup>Ac</sup> |
| Pre-heated conventional resin-based composite (Filtek Z350 XT) | 65.3 ± 3.2 <sup>Ac</sup>         | 60.6 ± 2.3 <sup>Bd</sup>                          | 55.5 ± 6.6 <sup>Cd</sup> | 45.0 ± 3.0 <sup>Dd</sup> |

Values are means ± standard deviations (n=6)

In % DC column, distinct capital letters indicate significant difference among the different ceramic translucencies for each luting composite while distinct lowercase letters indicate significant difference among the luting composites for each ceramic translucency ( $\alpha=0.05$ ). Significance values were:  $p < 0.001$  for the luting composite,  $p = 0.010$  for the ceramic translucency, and  $p = 0.001$  for the interaction between the luting composite and the ceramic translucency

## Results

The obtained *TP* values were 16.4 for HT, 13.4 for LT, and 12.6 for MO. The correspondent  $L^*a^*b^*$  color space data are presented at Table 2. Table 2 also demonstrates the information provided by the analyses performed with the power meter. The light irradiance value obtained with direct exposure to the sensor was 1218 mW/cm<sup>2</sup> and reduced to 810, 692 and 566 mW/cm<sup>2</sup> with the interposition of HT, LT and MO ceramic discs, respectively.

The DC values are described in Table 3. Considering the ANOVA evaluation, the lithium-disilicate translucency effect over the final DC was not statistically significant ( $p = 0.112$ ). On the other hand, the effect of the resin-based luting composite ( $p < 0.001$ ), as well as the interaction between these two factors ( $p = 0.002$ ) were statistically significant. Considering the influence of the different lithium-disilicate translucencies over DC values, no significant differences among the HT, LT, and MO were observed for all materials except for P-HRBC ( $p < 0.001$ ). Significantly different DC values were observed among the luting composite materials. For all different lithium-disilicate translucencies ( $p < 0.05$  for LT, HT, and MO), the D-AC showed the highest values, followed for the P-AC, then by the FRBC, and then by the P-HRBC, which presented the lowest values.

## Discussion

For all ceramic restorations, the final physical properties of the resin-based luting material may determine the clinical success [24]. Therefore, in theory, a low monomer–polymer conversion could correspond to reduced mechanical and color stabilities, ultimately leading to premature clinical failures. Considering the analysis of variance results the DC was not dependent on the lithium-disilicate translucency and, consequently, the



research hypothesis was rejected. However, it is important to mention the exception observed with the pre-heated conventional resin-based composite.

It should be expected that the ceramic translucency would directly affect the degree of C=C conversion [25] but this was not a general trend in the current study. This could be explained by the fact that the ceramic samples thickness was reduced and therefore, the optical differences among those materials were not enough to promote distinct light attenuation or not within a scale that provides distinct data. Another explanation could be related to the fact that a very powerful LED light curing unit was used and sufficient energy was delivered to the tested materials besides the effect of light attenuation. As seen in Table 1, at least 566 mW/cm<sup>2</sup> was able to achieve the luting materials in the worst case scenario (with the MO translucency), and by using 40 s of light exposure it was enough to provide more than 22 J/cm<sup>2</sup>. These outcomes corroborate with Faria-e-Silva and Pfeifer [10] that performed an in vitro study to determine the effectiveness of high-power LEDs to polymerize visible light-activated resin cement through ceramics and concluded that the ceramic interposition had only a slight effect on polymerization kinetics. In this study, the authors also detected that the ceramic interposition had some effect over the final DC only when ceramic discs with 1.5 mm thickness were used. It is also important to remember that only lithium-disilicate-based ceramics were tested in the current study and that similar behavior could not be expected when other ceramic types or even indirect composites are used. In example, recent studies have demonstrated that the zirconia type might influence the final DC of some luting agents as well as the consequent properties [26–28].

Pre-heated regular composites have been suggested for different clinical applications, such as to improve the handling characteristics for direct restorations or to become fluid enough to serve as a proper luting agent [29, 30]. In vitro investigations have demonstrated that pre-heated composites might have some benefits. Gresnigt et al. [20] found that luting of lithium disilicate laminate veneers by pre-heated regular composite (Enamel HFO, Micerium) resulted in higher survival and fracture resistance than a regular resin cement (Variolink Esthetic LC, Ivoclar Vivadent). This behavior could be related to the filler content, which is higher for regular composites. Additionally, greater conversion of monomers could be obtained and requiring reduced light exposure than with room-temperature composites due to greater mobility at the initial stages [31]. Such behavior was not observed in the current study and the DC of pre-heated regular composite was affected by the ceramic interposition and at this point it is important to clarify that the rheological behavior of pre-heated composites is material-brand dependent [32].

The current investigation considered the use of Filtek Z350 XT (Filtek Supreme XT in some countries) to keep the same manufacturer from the resin cements and, therefore, to reduce part of the variations resultant from large differences that exists among the formulations from different companies. Al-Ahdal et al. [32] analyzed the rheological properties of resin composites according to variations in composition and temperature and demonstrated that Filtek Z350 XT exhibited the highest viscosity both at 25 °C and 37 °C. Considering the outcomes from the present investigation, it can be speculated that the very high content of Filtek Z350 XT makes it more dependent on the amount of reaching light due to the light scattering effect and might explain



why pre-heated composite was the only material affected by the ceramic translucency when considering the pairwise comparisons.

Although the current investigation brings important data for the current knowledge, it is important to consider some limitations and future directions:

- Besides the composite was pre-heated at  $68 \pm 1$  °C, the real temperature at the moment of the activation was probably much lower due to cooling effect caused by the contact both with the ceramic veneer and also the ATR crystal that were kept at room temperature as it took  $\approx 90$  s from the removal from the oven to the end of the FTIR reading. Although this represents a real clinical situation, the temperature reduction impairs proper molecular diffusion and, thus, degree of C=C conversion [33]. Consequently, further studies have now been conducted to determine the effect of pre-heating both different material brands and, also, the effect of the laminate veneer temperatures on the degree of C=C conversion.
- The current investigation considered the degree of C=C conversion after 5 min and it is crucial to determine the post-curing after a long period of time.
- Degree of conversion is an important aspect, but experiments aiming to analyze both the mechanical properties, stress development and the degradation resistance are crucial.

## Conclusions

Based on the results of this study it can be concluded that the degree of C=C conversion was not influenced by the different lithium-disilicate translucency for any luting agent, except for the pre-heated conventional resin-based composite which presented significant lower conversion.

## Abbreviations

C=C: Carbon double bond; DC: Degree of conversion; HT: High translucency; LT: Low translucency; MO: Medium opacity; TP: Translucency parameter; cm<sup>2</sup>: Square centimeter; mW: Milliwatt; mW/cm<sup>2</sup>: Milliwatt/square centimeter; D-AC: Dual-activated resin-based cement; P-AC: Photo-activated resin-based cement; FRBC: Flowable resin-based composite; P-HRBC: Pre-heated conventional resin-based composite; ATR: Attenuated total reflectance; FTIR: Fourier transform infrared; IR: Infrared spectrum; °C: Celsius; mm: Millimeter; LED: Light-emitting diode; J/cm<sup>2</sup>: Joules/square centimeter.

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## Authors' contributions

LFS conceived and designed this study. ASQ, RH and VS performed the experiments. RH, VS and LFS analyzed and interpreted the data. LSF, LC and WP reviewed the manuscript. All authors read and approved the final manuscript.

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## Availability of data and materials

All relevant data is presented in the manuscript and additional information can be made available on request if necessary.

## Competing interests

The authors declare that they do not have competing interests.

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