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Investigating the limits of resin-based luting composite photopolymerization through various thicknesses of indirect restorative materials

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Abstract

Objective

sTo determine the limitations of using light-curable resin-based luting composites (RBLCs) to bond indirect ceramic/resin-composite restorations by measuring light transmittance through indirect restorative materials and the resulting degree of conversion (DC) of the luting-composites placed underneath.

Methods

Various thicknesses (0–4 mm) and shades of LAVA Zirconia and LAVA Ultimate were prepared and used as light curing filters. A commercial, light curable RBLC, RelyX Veneer (control) was compared with four experimental RBLCs of the following composition: TEGDMA/BisGMA (50/50 or 30/70 wt%, respectively); camphorquinone/amine (0.2/0.8 wt%) or Lucirin-TPO (0.42 wt%); microfillers (55 wt%) and nanofillers (10 wt%). RBLCs covered with the LAVA filter were light-cured for 40 s, either with the dual-peak BluephaseG2 or an experimental device emitting either in the blue or violet visible band. The samples were analyzed by Raman spectroscopy to determine DC. Light transmittance through the filters was measured using a common spectroscopy technique.

Results

All the factors studied significantly influenced DC ($p \leq 0.05$). RBLCs with increased TEGDMA content exhibited higher DC. Only small differences were observed comparing DC without filters and filters ≤1 mm ($p \geq 0.05$). For thicknesses ≥2 mm, significant reductions in DC were observed ($p < 0.05$). Transmittance values revealed higher filter absorption at 400 nm than 470 nm. A minimal threshold of irradiance measured through the filters that maintained optimal DC following 40 s irradiation was identified for each RBLC formulation, and ranged between 250–500 mW/cm^2.

Significance

This work confirmed that optimal photopolymerization of RBLCs through indirect restorative materials (≤4 mm) and irradiation time of 40 s is possible, but only in some specific conditions. The determination of such conditions is likely to be key to clinical success, and all the factors need to be optimized accordingly.

1 Introduction

Clinical studies describe high performance of bonded ceramic restorations (esthetics, good survival rate), not only to restore anterior teeth [1–4], but also for extensive posterior restorations [5–7]. For both indications, the bonding quality is essential to provide clinical effectiveness, especially for partial restorations. Weaknesses in the bonding interface may lead to early clinical failures; mainly loss or fracture of the restoration, but also possibly favor the occurrence of other issues such as secondary caries, post-operative sensitivity or marginal discolouration due to marginal leakage [8].

Traditionally, a dual-cure resin-based luting composite (RBLC) is preferred for the placement of indirect restorations, to ensure effective polymerization even through thick and/or opaque restorations. The dual-cure chemistry supposedly combines the assurance of 'dark' chemical curing with some of the numerous advantages provided by purely light-curable systems. The latter notably include improved handling aspects, such as a single paste without the need for mixing, better control of working time, faster setting, easier excess removal or improvement of the interface colour stability [9]. Despite these advantages, very few works investigated the use of purely light-curable composites to lute indirect restorations [10,11]. One reported the possibility to reach an "adequate" polymerization of a conventional resin composite (described as 80% of the maximum material microhardness) when light cured through 7.5 mm thick 'endocrowns' [11]. Another revealed higher bond strength values when light curable resin composites were used to lute 4 mm thick inlays compared with the use of a dual-cure resin composite [10]. Such observations may be explained by two major elements: firstly, light curable resin composites usually contain more fillers than dual cure resin cements [12], hence higher intrinsic mechanical properties [10]. Secondly, photopolymerization processes probably generate a higher concentration of free radicals, which can be profitable during the autoacceleration step in dimethacrylate resins. During such step, any new growth centre created indeed leads to efficient chain propagation since the low mobility of the building polymer chains reduces the likelihood of bimolecular termination [13]. This reinforces the potential interest of utilizing solely light-curable chemistries not only to lute veneers [14] or thin inlays, but also thicker posterior restorations, such as endocrowns [10,11]. The importance of effective photopolymerization in light-curable RBLCs, and even those systems that include autopolymerization chemistries, is highlighted in numerous works [15–18]. For example, a previous study has reported a three-fold decrease in microhardness of dual-cure RBLCs when light cured through thick (4 mm), compared with thinner (2 mm or less), or no use of indirect ceramic filters [15]. A similar observation was made when measuring the degree of conversion of a dual-cure RBLC, with a two- to four-fold decrease of conversion through opaque 2 mm ceramic filters [17]. The autopolymerization step in a dual-cured system seems therefore insufficient to ensure optimal polymerization of luting composites. Hence, undercuring of dual-cure materials beneath thick indirect restorations remains a risk, which is potentially worsened with systems that use light-curable chemistries alone. Effective polymerization of the latter, is indeed necessary to ensure optimal physico-mechanical properties [13,19] and colour stability [20,21], thereby reducing the risk of interfacial failure.

Light transmittance through a tooth-coloured indirect restoration is significantly affected by material type. Veneers are commonly fabricated with feldspathic glass (porcelain), which exhibit relatively high translucency, however, more opaque materials exist, especially those fabricated using more modern CAD/CAM processes, including resin-based composites, particle reinforced ceramic composites (e.g. lithium disilicates and leucite-based ceramics) and polycrystalline ceramics (e.g. alumina and zirconia), the latter of which are expected to be the least translucent (notwithstanding modern attempts to increase translucency of monolithic polycrystalline crowns by adjusting the phase stabilisation dopant, grain size, and so forth). Therefore, if light transport is limited by the opacity of the indirect material, other inherent material chemistries that circumvent the need for effective polymerization using higher irradiance is certainly worthy of consideration.

The interest of using alternative photoinitiator systems to the classical combination of camphorquinone/amine (CQ), such as Type 1 acylphosphine oxides, has been extensively described for direct restorative resin composites. Notably, higher final DC and higher mechanical properties have been reported using curing times shorter than 3 s [22–26]. This was explained by higher molar absorptivity and quantum yield efficiency [27,28], which are potentially key aspects as regards light-curing through indirect materials. Indeed, low transmittance is iexpected through thick indirect material layers, which explains the relatively long irradiation times that were used when luting with light-activated (non-dual cure) resin-composites (from 40 s [10] to several cycles of 90 s [11]).

Consequently, the aim of this work was to determine the limits of RBLC photopolymerization by measuring light transmittance through various thicknesses of indirect restorative materials and the resulting degree of conversion (DC) of the resin cements placed underneath. Experimental RBLCs of various monomer ratios and photoinitiator content, as well as filters of different materials and shades were considered.

2 Materials and ~~M~~methods

The thickness of large indirect restorations such as overlays or endocrowns is inhomogeneous (Fig. 1A, B). In order to experimentally model such variability of thickness in a reproducible manner, various CAD/CAM blocks were used to prepare 10 mm diameter disc-shaped filters of 4 different thicknesses: 0.5, 1, 2 and 4 mm (±0.01 mm).

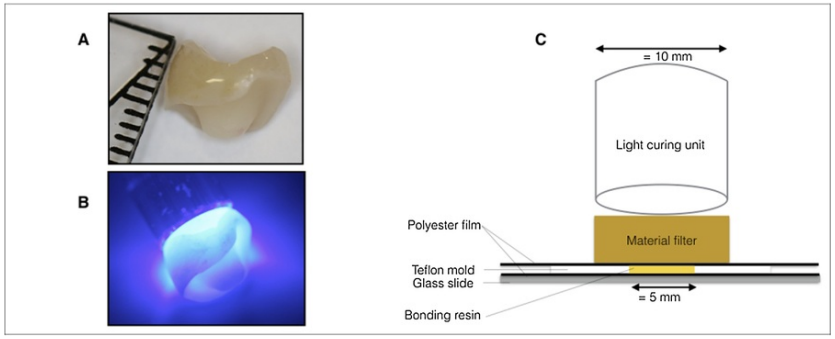

Fig. 1 Example of Lava Ultimate ~~E~~(3M-ESPE) overlay of variable thickness (A), and the same overlay irradiated with BluePhaseG2 (Ivoclar-Vivadent) (Please add (B)); (C) experimental setup to polymerize RBLC through a restorative material filter (thicknesses ranging from 0.5 to 4 mm).

alt-text: Fig. 1

The CAD/CAM blocks were made of either a polycrystalline, yttria-stabilised zirconia ceramic, LAVA-Zr (shades A3 and uncoloured ~~–~~ Zr-A3 and Zr-U) or a resin composite block with dispersed fillers, LAVA Ultimate (shades A3 and MC2~~ Ult-A3 and Ult-MC2 –~~ Ult-A3 and Ult-MC2 – the latter reported as more opaque) (3M-ESPE, St Paul, MN, USA). Five RBLCs were light-cured through these filters: four experimental formulations and RelyX Veneer (3M-ESPE, St Paul, MN, USA), a commercially-available light-curable RBLC used as control. Experimental formulations were prepared to mimic the commercial material (Table 1). The experimental formulations contained two proportions of conventional monomers TEGDMA and BisGMA in ratios of 50/50 and 30/70 wt%, respectively. Each resin blend contained either camphorquinone/amine (0.2/0.8 wt% ~~–~~ CQ) or Lucirin-TPO (0.42 wt% ~~–~~ Lu-TPO) as the photoinitiator. The different components were weighed using an electronic analytical balance (AND FR-300-MKII, A&D INSTRUMENTS LIMITE, Abingdon, U.K. ~~–~~ accuracy ±100 μg) and placed sequentially in opaque plastic pot to prevent light exposure. Barium glass microfillers and fumed silica nanofillers were added in amounts of 55/10 wt%, respectively. Silanated fillers were used, both for the nano- and micro-scale particles.

Table 1 Composition of resin-based composite cements.

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Resin	Fillers	Monomers	Photo initiator
~~RelyX Veneer(3~~ RelyX Veneer (3M ESPE, St Paul, MN, USA)	Silane treated ceramic (55–65 wt%),	~~Silane treated silica (1–10 wt%) and~~BisGMA 10–20% of total weight	Titanium dioxide <1 wt%)
	Silane treated silica (1–10 wt%) and	TegDMA 10–20% of total weight	Ethyl 4-dimethyl aminobenzoate (EDMAB) (<1 wt%)
	Reacted polycaprolactone polymer (1–10 wt%)	*	B~~isGMA 10-20% of total weightTegDMA 10-20% of total weight*Titanium Dioxide <1 wt%)Ethyl 4-Dimethyl Aminobenzoate (EDMAB) (< 1 wt%)Benzotriazol(< 1%wt)Diphenyliodonium Hexafluorophosphate (< 1 wt%)*~~enzotriazol (<1 wt%)
	*		Diphenyliodonium hexafluorophosphate (<1 wt%)
			*
CQ50/50	Barium glass fillers silanated (G018-186/K6, d50 = 3 ± 1 µm, Schott AG, Landshut Germany) and methacrylsilane treated fumed silica (12 nm, AEROSIL® R 7200 Aerosil 7200, Evonik Industries, Germany) in amounts of 55/10 wt% respectively.	50/50 wt% of Bis-GMA and TegDMA resin (Sigma–Aldrich)	Camphorquinone (Sigma Aldrich, CAS Number 10334-26-6) as the photoinitiator and dimethylaminoethyl methacrylate (Sigma Aldrich) as co-initiator, in the proportions of 0.2/0.8 wt%
CQ30/70		70/30 wt% of Bis-GMA and TegDMA resin (Sigma–Aldrich)	

TPO 50/50		50/50 wt% of Bis-GMA and TegDMA resin (Sigma–Aldrich)	Lucirin-TPO (TPO, from BASF) 0.42 wt% as the photoinitiator
TPO 70/30		70/30 wt% of Bis-GMA and TegDMA resin (Sigma–Aldrich)	

*~~a~~According to manufacturers informations.

Bis-GMA: Bisphenol A glycerolate dimethacrylate, CAS Number: 1565-94-2.

TEGMA: Triethylene Glycol Dimethacrylate, CAS Number 109-16-0.

Fillers were incorporated sequentially using a dual asymmetric centrifuge (Speed mixer, FlackTek, USA) for 30 ~~second~~s at 3500 rpm for the nanofillers, and at 2500 rpm for the microfillers. The mixing procedure (rpm, time, etc.) was previously optimized in other work [25].

Light sources were either the dual-peak BluephaseG2 (BPG2, Ivoclar-Vivadent, Schaan, Liechtenstein; curing tip diameter = 10 mm; “High power”) or a light-curing device (AURA, Lumencor, USA; curing tip diameter = 6 mm) emitting either in the blue (AURAblue; 455–485 nm) or in the violet (AURAviolet; 395–415 nm); the irradiance for both spectral outputs was calibrated and set at around 1000 mW/cm^2. The irradiance values were measured with the Thorlabs Optical Power and Energy Meter PM100USB at 1020 mW/cm^2 for AURAviolet, 1030 mW/cm^2 for AURAblue and 1119 mW/cm^2 for the BPG2. The relationship between the absorption spectra of the photoinitiators (CQ and Lu-TPO) and the curing lights emission spectra were compared (Fig. 2; [24]).

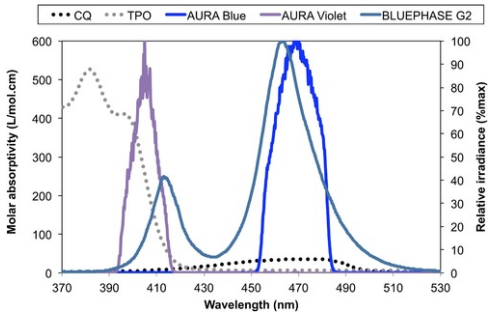

Fig. 2 Emission spectrum of investigated curing lights (presented in relative irradiance, 100% being the maximum spectral irradiance), compared to the molar absorptivity of the two photoinitiators included in the investigated materials, i.e. Lu-TPO and CQ. The dotted lines refer to the left-Y-axis (Molar absorptivity) and the plain lines to the right-Y-axis (Relative irradiance).

alt-text: Fig. 2

Light transmittance was measured through the various filters using a UV–vis spectrometer (USB4000, Ocean Optics, UK; n = 3). The spectrometer was coupled to a 200 μm optical fibre and an opaline glass CC3 cosine corrector (3.9 mm diameter of collection area, Ocean Optics, UK) and calibrated with a National Institute of Standards and Technology (NIST) traceable light source (Mikropack DH2000/Ocean Optics, UK). Following calibration, the integration time was set automatically with a boxcar width of 0 and spectra average equals 1. The LAVA filters were interposed centrally between the tip of the light curing unit and the cosine corrector. The light device was fixed in a standardized position, with the surface of the tip parallel and in contact with the filter surface and the filter parallel and as close as possible to the cosine corrector. The absolute irradiance was calculated as the integral beneath the curve.

The light transmittance was measured with and without a polyester film, and the transmittance profile showed that there was no significant difference ($p < 0,05$).

The RBLC were placed in 1 mm thick, 5 mm diameter Teflon molds, covered on each side with a polyester film (≅0.1 mm thick), compressed between two glass slides to extrude excess, and covered by a ceramic filter through which 40 s light irradiation was performed with the light-tip parallel and in direct contact (Fig. 1C).

After photopolymerization, the samples (n = 3) were stored ‘dry’, in the dark at room temperature for one week, before being analyzed by Raman spectroscopy (DXR Raman Microscope, Thermo Scientific, Madison, WI USA) to

determine the degree of conversion (DC, in %) [29] on the upper RBLC surface (n = 3). Briefly, a frequency stabilized single mode diode laser excited the samples through a 50× microscope objective. The spectra were acquired in the area of 1600 cm^{-1}, using a 50 slit, a 60 s irradiation, 5 accumulations, and a grating of 400 lines/mm. The calculation of DC was based on the decrease in intensity of the peak corresponding to the methacrylate C=C group at 1640 cm^{-1} compared with an unpolymerized sample. The aromatic peak at 1610 cm^{-1} was used as the internal standard [30]. Given the small thickness of the RBLC layer (around 25 µm [31]), it was assumed that the DC measurement at the upper RBLC surface of the 1 mm thick samples was representative of the conversion of the whole RBLC layer.

Statistical analyses were performed with the JMP Pro 12 software (SAS). One-way ANOVA were performed followed by multiple comparisons with a level of significance of p = 0.05; when normal distribution of the data could not be verified, the non-parametric Wilcoxon test was used; when normality was verified, HSD Tukey's test was used.

3 Results

An inverse logarithmic relationship was observed between transmittance and filter thickness. After logarithmic transformation of transmittance, linear correlations with correlations coefficients between 0.89 and 0.97 were observed (Fig. 3). Fig. 3 also confirms the previous affirmation that the transmittance is significantly lower for AURAviolet than for BPG2 and AURAblue, especially for Zr-A3. Light transmittance for all the materials are relatively similar between AURAblue and BPG2, although transmittance is generally higher for BPG2.

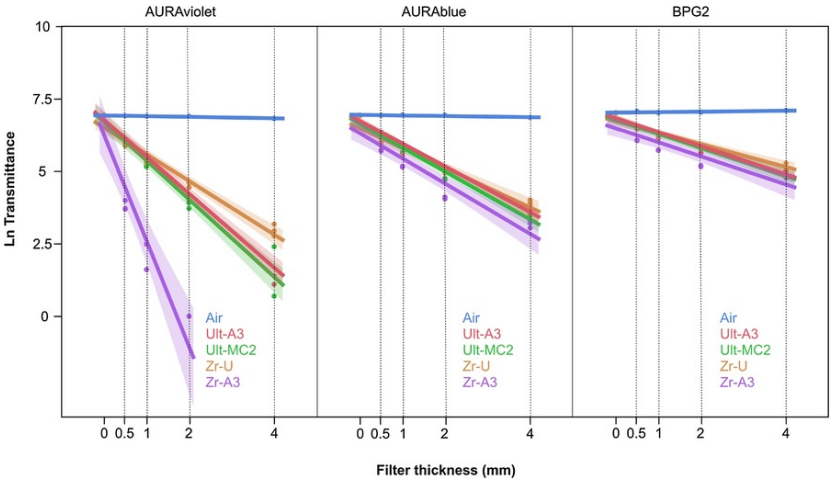

Fig. 3 Transmittance (Ln transformation) as a function of filter thickness, for the different lights (AURAblue in two different wavelengths and BPG2). For all four types of filters, linear correlations were drawn, and are associated with the following correlation coefficients: AURAviolet Ult-A3 (R^2 = 0.97), Ult-MC2 (R^2 = 0.96), Zr-A3 (R^2 = 0.91), Zr-U (R^2 = 0.96); AURAblue Ult-A3 (R^2 = 0.98), Ult-MC2 (R^2 = 0.97), Zr-A3 (R^2 = 0.89), Zr-U (R^2 = 0.92); BPG2 Ult-A3 (R^2 = 0.96), Ult-MC2 (R^2 = 0.93), Zr-A3 (R^2 = 0.80), Zr-U (R^2 = 0.90). For all transmittance data, standard deviations can be found in Table 2.

alt-text: Fig. 3

Light transmittance through the ceramic filters was significantly affected by thickness ($p < 0.0001$), type, shade ($p = 0.0205$ for LAVA-Zr) as well as by light type ($p < 0.0001$), but not for either shade of Ultimate ($p = 0.4218$) (Wilcoxon test) (representative examples in Fig. 3, full results in Table 2)

Table 2 Light transmittance depending on filter thickness, in % of maximum transmittance (without interposition of any filter)*.

alt-text: Table 2

Curing light	Filter material	0.5mm	1mm	2mm	4mm
BPG2	Ult-A3	63.54 (0.71) A a	47.72 (0.59) B a	25.17 (0.21) C b	13.52 (0.22) D b
	Ult-MC2	62.64 (1.00) A a	40.60 (0.21) B c	22.58 (0.27) C c	13.28 (0.25) D b
	Zr-A3	38.40 (0.52) A c	27.23 (0.27) B d	15.76 (0.31) C d	10.90 (0.41) D c
	Zr-U	56.72 (0.14) A b	41.94 (1.01) B b	28.95 (0.41) C a	17.51 (0.25) D a
AURAblue	Ult-A3	54.75 (1.07) A a	33.12 (0.31) B a	14.04 (0.30) C a	4.01 (0.39) D b
	Ult-MC2	47.44 (3.61) A b	27.42 (0.48) B b	11.00 (0.30) C b	3.27 (0.15) D c
	Zr-A3	29.04 (0.50) A d	16.72 (0.22) B d	5.72 (0.19) C c	2.36 (0.29) D d
	Zr-U	37.41 (2.60) A c	24.35 (0.35) B c	13.65 (0.11) C a	4.95 (0.40) D a
AURAviolet	Ult-A3	43.86 (0.31) A a	22.06 (0.29) B a	6.11 (0.62) C b	0.75 (0.71) D b
	Ult-MC2	41.04 (2.32) A b	17.25 (0.29) B c	4.77 (0.69) C c	0.42 (0.57) D b
	Zr-A3	4.41 (0.76) A d	0.33 (0.94) B d	**	**
	Zr-U	35.29 (0.45) A c	19.48 (0.40) B b	9.31 (0.85) C a	1.93 (0.39) D a

*Similar upper-case letters and various shades of grey connect in the same row results which are not significantly different (based on Tuckey's test, p = 0.05). Lower-case letters connect in the same column (and for a given curing light) results which are not significantly different at a given filtre thickness (based on Tukey's test p = 0.05). **No transmittance value could be measured.

Overall, light transmittance decreased significantly ($p < 0.05$) with each additional ceramic filter thickness (Table 2). The transmittance was generally low through all 4 mm thickness filters, i.e. between 10.9 and 17.5% for BPG2, between 2.4 and 5.0% for AURAblue, and between 0.42 and 1.93 for AURAviolet (Fig. 3 and Table 2).

The comparison of light transmittance between the ceramic and resin composite of similar shade (A3) revealed a lower transmittance at each thickness for each material, respectively (purple and red curves, respectively, in Fig. 3; Table 2) for each of the curing lights.

The effect of filter shade was more obvious for LAVA-Zr, with a significantly lower transmittance through Zr-A3 than Zr-U at each thickness (purple and orange curves, respectively, in Fig. 3) and for all three lights (Table 2). For LAVA-Ult, a tendency of higher transmittance was observed for Ult-A3 as compared to Ult-MC2, but the differences were not statistically significant in all conditions (red and green curves, respectively, in Fig. 3; Table 2).

BPG2 seemed to be associated with higher percentage of transmittance than AURA for each thickness and material type. The same observation could be done for AURAblue as compared to AURAviolet, the latter yielding the lowest transmittance values (Table 2).

Regarding DC, the type of RBLC ($p = 0,0001$, Wilcoxon test), photoinitiator and monomers contents ($p < 0,0001$), filter type ($p = 0,0005$), filter shade ($p = 0,0061$ for LAVA-Zr) and light ($p < 0,0001$) as well as thickness ($p = 0,0001$) all significantly influenced the values, except for the shade of Ultimate ($p = 0.5349$) (Wilcoxon test).

In order to identify the relationship between light transmittance and RBLC conversion, DC was plotted against transmittance (Fig. 4). Thereby, it is possible to identify the transmittance threshold necessary to maintain optimal DC after 40 s irradiation for each RBLC formulation and curing light. For all conditions, DC curves inflection was located between 250 and 500 mW/cm^2.

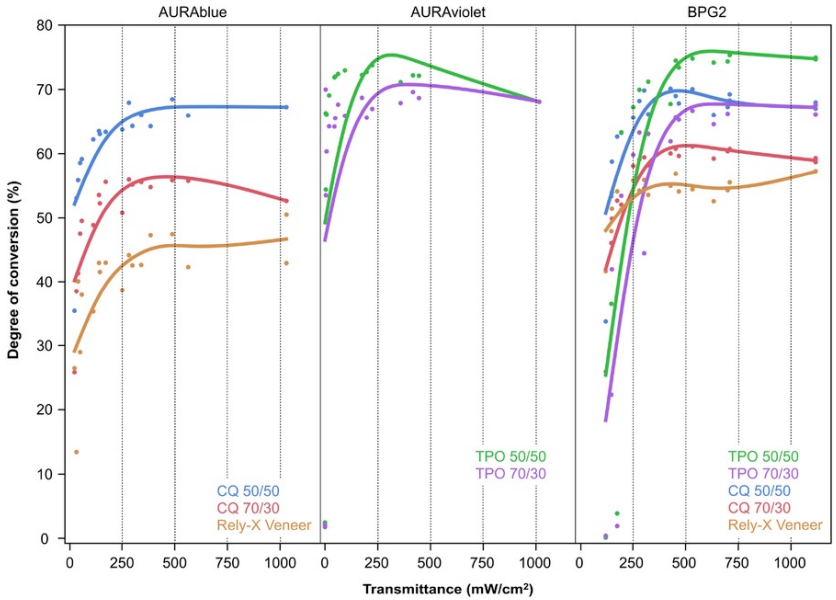

Fig. 4 Degree of conversion (%) in relation with the measured transmittance (mW/cm^2); smoothed splined curves with lambda = 0.1). Comparison of RBLC compositions for each curing light. For all DC data, the standard deviations are in Table 3, 4 and 5s 3–5.

alt-text: Fig. 4

For experimental CQ compositions, BPG2 and AURAblue are comparable for CQ50/50, and BPG2 was slightly more efficient for CQ70/30 (blue and red curves, respectively, in Fig. 4). For experimental Lu-TPO-based materials, AURAviolet is more efficient at each level of transmittance than BPG2 (green and purple curves, respectively; Fig. 4). For the commercial product, Rely-X Veneer, BPG2 yields much higher DC at each level of transmittance (orange curve in Fig. 4).

When comparing curing lights efficiency for each RBLC (Fig. 4), it appears that BPG2 and AURAblue have relatively comparable efficiencies in experimental CQ-based materials, while BPG2 is clearly more efficient for the supposedly CQ-based Rely-X Veneer. For Lu-TPO-based materials, AURAviolet appears as more efficient than BPG2 at low irradiance.

Without any filter, the DC values ranged between 57.2 and 74.7%. DC of Lu-TPO-RBLC was significantly higher that CQ-based one at similar monomer ratio ($p < 0.05$). DC of 50/50 TEGDMA/Bis-GMA was significantly higher than DC of the 70/30 ratio for a similar photoinitiator system. The lowest DC was observed for the commercial control material Rely-X Veneer (Fig. 4 and Tables 3–5).

Table 3 Degree of conversion depending on filter thickness for Bluephase G2*.

alt-text: Table 3

Filter material	Resin composition	No filter	0.5mm	1mm	2mm	4mm
Ult-A3	CQ 50/50	67.9 (0.8) Ab	69.17 (0.68) A b	69.97 (0. 75) A b	68.13 (0.40) A a	58.67 (0.93) B a
	CQ 70/30	58.9 (0.8) Ac	60.63 (1.15) A c	61.03 (0.86) A d	59.00 (1.15) A c	49.77 (1.86) B c
	TPO 50/50	74.7 (0.6) Aa	72.27 (0.49) A a	74.73 (0.75) A a	69.87 (0. 72) B a	53.87 (2.10) C b
	TPO 70/30	67.1 (0.9) Ab	68.20 (0.96) A b	66.60 (0.50) AB c	63.20 (2.00) B b	41.90 (1.82) C d
	RelyX Veneer	57.2 (1.6) Ac	55.42 (0.48) A d	54.34 (0.77) AB e	54.12 (1.40) AB d	51.32 (0.82) B bc
Ult-MC2	CQ 50/50	67.2 (0.8) Ab	67.18 (1.10) A b	68.90 (1.00) A b	65.54 (0.54) A a	53.25 (1.38) B a
	CQ 70/30	58.9 (0.8) Ac	60.28 (0.52) A c	60.69 (0.56) A d	58.00 (0.31) A bc	45.98 (1.62) B a
	TPO 50/50	74.7 (0.6) Aa	74.27 (1.40) A a	74.41 (0.98) A a	67.16 (0.56) A a	36.50 (6.88) B b
	TPO 70/30	67.1 (0.9) Ab	66.12 (0.26) A b	65.59 (1.70) A c	59.73 (2.88) A b	22.38 (7.88) B c
	RelyX Veneer	57.2 (1.6) Ac	54.20 (1.14) A d	56.77 (1.00) A e	55.71 (1.38) A c	47.84 (1.33) B a
Zr-A3	CQ 50/50	67.2 (0.8) Ab	70.03 (0.75) A a	69.77 (0.74) A a	62.57 (0.81) B a	33.77 (2.45) C b
	CQ 70/30	58.9 (0.8) Ac	59.93 (0.40) A b	59.33 (0.57) A b	52.57 (3.51) B b	25.97 (4.00) C c
	TPO 50/50	74.7 (0.6) Aa	67.67 (1.89) B a	54.47 (3.84) C c	3.90 (0.173) D c	0.10 (0.09) D d
	TPO 70/30	67.1 (0.9) Ab	61.83 (2.35) B b	44.40 (0.95) C d	1.9 (1.85) D c	0.37 (0.54) D d
	RelyX Veneer	57.2 (1.6) Ac	54.88 (1.77) A c	55.79 (1.92) A bc	54.01 (0.69) A b	41.61 (1.25) B a
Zr-U	CQ 50/50	67.2 (0.8) Ab	65.94 (6.90) A b	67.74 (0.43) A b	66.06 (0.47) A b	63.15 (4.61) A a
	CQ 70/30	58.9 (0.8) Ac	59.14 (0.69) A c	59.55 (0.59) A d	58.11 (0.52) A d	51.94 (0.53) B b
	TPO 50/50	74.7 (0.6) Aa	74.10 (1.05) A a	73.34 (0.37) AB a	71.13 (1.11) B a	63.23 (0.98) C a
	TPO 70/30	67.1 (0.9) Ab	64.50 (1.34) AB bc	65.21 (0.87) AB c	62.99 (0.47) B c	53.33 (0.84) C b
	RelyX Veneer	57.2 (1.6) Ac	52.47 (3.04) AB d	54.01 (0.88) AB e	53.47 (1.60) AB e	51.17 (0.64) B b

A1

*Similar upper-case letters and various shades of grey connect in the same row results which are not significantly different (based on Tuckey's test, p = 0.05). Lower-case letters connect in the same column results which are not significantly different at a given filtre thickness (based on Tukey's test p = 0.05).

Annotations:

A1. Please suppress this line in the middle of the box

A2. This thick line should not be there. Please change with tables attached to your querie

Table 4 Degree of conversion depending on filter thickness for AURA blue (468 nm+-10 nm)*.

alt-text: Table 4

Material filter	Resin composition	No filter	0.5mm	1mm	2mm	4mm
Ult-A3	CQ 50/50	66.7 (1.1) Aa	65.87 (0.46) A a	65.93 (1.02) A a	62.97 (0.25) A a	55.77 (2.45) B a
	CQ 70/30	52.5 (1.7) Ab	55.66 (1.08) A b	55.48 (2.38) A b	52.15 (4.08) A b	41.29 (3.79) B b
	RelyX Veneer	46.6 (4.0) Ac	42.25 (1.45) A c	42.58 (0.76) A c	41.48 (1.88) A c	40.02 (3.09) A b
Ult-MC2	CQ 50/50	66.7 (1.1) Aa	68.40 (1.73) A a	67.87 (1.02) A a	62.13 (1.65) B a	52.97 (1.50) C a
	CQ 70/30	52.5 (1.7) ABb	55.75 (1.68) A b	55.87 (0.48) A b	48.77 (2.23) B b	38.48 (2.88) C b
	RelyX Veneer	46.6 (4.0) Ab	47.37 (1.10) A c	44.10 (0.36) A c	35.33 (0.45) B c	13.43 (5.06) C c
Zr-A3	CQ 50/50	66.7 (1.1) Aa	64.23 (0.81) AB a	63.30 (0.87) B a	59.07 (0.75) C a	35.43 (0.70) D a
	CQ 70/30	52.5 (1.7) Ab	55.09 (0.56) A b	55.50 (0.81) A a	49.43 (5.36) A b	25.97 (4.00) B b
	RelyX Veneer	46.6 (4.0) Ac	42.51 (0.95) A c	42.93 (6.15) A b	37.94 (6.77) A b	26.50 (0.88) B b
Zr-U	CQ 50/50	66.7 (1.1) Aa	64.20 (0.87) B a	63.67 (1.27) B a	63.43 (0.45) B a	58.46 (0.63) C a
	CQ 70/30	52.5 (1.7) Ab	54.68 (0.73) A b	50.68 (7.06) A b	53.45 (0.79) A b	47.44 (0.50) A b
	RelyX Veneer	46.6 (4.0) Ab	42.70 (1.51) B c	38.63 (0.76) B c	42.90 (1.56) B c	29.00 (3.96) C c

*Similar upper-case letters and various shades of grey connect in the same row results which are not significantly different (based on Tuckey’s test, p = 0.05). Lower-case letters connect in the same column results which are not significantly different at a given filtre thickness (based on Student t test, p = 0.05).

Table 5 Degree of conversion depending on filter thickness for AURAviolet (400 nm+-10 nm).

alt-text: Table 5

Material Filter	Resin composition	No filter	0.5mm	1mm	2mm	4mm
Ult-A3	TPO 50/50	67.8 (0.9) Ba	72.06 (0.36) A a	72.69 (0.74) A a	72.33 (2.15) A a	66.03 (1.48) B a
	TPO 70/30	68.2 (0.3) Aa	68.59 (0.28) A b	66.60 (0.50) A b	67.56 (1.05) A b	60.26 (2.06) B b
Ult-MC2	TPO 50/50	67.8 (0.9) Aa	72.71 (0.83) A a	72.18 (0.53) A a	71.93 (1.29) A a	54.29 (3.48) B a
	TPO 70/30	68.2 (0.3) Aa	69.52 (2.98) A a	68.62 (0.54) A b	65.45 (1.12) A b	53.41 (2.15) B a
Zr-A3	TPO 50/50	67.8 (0.9) Aa	71.81 (0.99) A a	66.21 (0.14) A a	0* B a	0* B a
	TPO 70/30	68.2 (0.3) A a	64.16 (2.10) A b	63.91 (1.20) A b	2.05 (0.72) B a	1.76 (0.06) B a
Zr-U	TPO 50/50	67.8 (0.9) Ba	71.06 (2.26) A a	72.65 (1.19) A a	72.88 (2.85) A a	69.00 (0.95) A a
	TPO 70/30	68.2 (0.3) Aa	67.81 (0.42) A a	65.53 (1.95) A b	65.81 (0.43) A b	64.18 (0.80) B b

Mean degree of conversion for the light-curing unit Aura. 400 nm. expressed in percents. The results in a same raw, unconnected with the same letter are significantly different (p-value 0.05), from the test Kruskal-Wallis. Minuscule letters show significantly difference between different resin composition. within a same material (p-value 0.05. from the Student's t-test).

*0 is not a measured value, because the DC was so low than we could not measure it.

While the effect of monomer ratio is important on the absolute DC value at a given filter thickness, DC values with increasing filter thickness and a given photoinitiator system decrease similarly for 50/50 and 70/30 ratios (Tables 3–5). Notably, the drops corresponding to significant differences can be observed at similar filter thicknesses.

Although DC without filters and with thin filters were higher for Lu-TPO-based RBLC, the trend reversed when using thick (4 mm) filters with BPG2 (Table 3). When using AURA however, where irradiance was comparable between violet and blue peaks, such differences were not observed, DC of Lu-TPO-based materials remaining higher at 4 mm, except for Zr-A3 (Tables 4 and 5).

Regarding filter type, as observed for transmittance, the comparison of DC between ceramic and resin composite filters was achieved for similar shade (A3), with a significantly lower DC for Zr-A3 than for Ult-A3 (Tables 3–5) for each of the three curing lights.

In relation to what was observed for transmittance, the effect of filter shade on DC was more obvious for LAVA-Zr, with a significantly lower DC through Zr-A3 than Zr-U and for all three lights (Tables 3–5). For Lava-Ult DC was higher through Ult-A3 than through Ult-MC2 (Tables 3–5), although differences were not statistically significant ($p > 0.05$).

Regarding the same shade for the two different materials, a significant difference was observed between Zr-A3 and Ult-A3 for the DC obtained through thicknesses >2 mm.

With regard to thickness, the use of filters ≤1 mm resulted in few significant differences in DC when compared with RBLCs without filters. For thicknesses ≥2 mm, more significant reductions in DC were observed, particularly at 4 mm, depending on the ceramic filter/light/photoinitiator combination. In general, the critical decrease in DC was observed between 2 and 4 mm. However, for some combinations, no significant decrease in DC was observed, even for 4 mm-thick filters ($p > 0.05$), i.e. Rely-X Veneer ~~– AURAblue – Ult-A3, CQ70/30 AURAblue – Zr-U, CQ50/50 BPG2 –~~ – AURAblue – Ult-A3, CQ70/30 – AURAblue – Zr-U, CQ50/50 – BPG2 – (Please replace by: i.e. Rely-X Veneer cured by AURAblue through Ult-A3; CQ70/30 cured by AURAblue through Zr-U; CQ50/50 cured by BPG2 through Zr-U.)Zr-U (Tables 3–5).

4 Discussion

The current work confirmed that optimal photopolymerization of RBLCs through indirect restorative materials (≤4 mm) and irradiation time of 40 s is possible, but only for specific conditions. The determination of such

conditions is likely to be key to clinical success, and all the factors studied in the present work (filter material type, thickness and shade, monomer composition, photoinitiator content, etc.) significantly impacted both transmission and conversion.

The first obvious limitation of this procedure was to achieve sufficient light transmittance for optimal polymerisation of the RBLC through the indirect restoration. The significant impact of material shade on transmittance observed here confirmed the findings of previous studies, i.e. that darker shades led to lower transmittance both in ceramics and resin composites [17,32,33]. This in turn resulted in lower conversion or lower mechanical properties of RBLC [16–18].

The inverse logarithmic relationship between material thickness and transmittance described in other works [32] was confirmed here (Fig. 3), the slope being specific to each curing light/material combination. The most important decrease in transmittance observed at shorter wavelengths (AURAviolet) with the investigated materials is also in accordance with previous work [34], and may represent a limitation for these systems. However, this may be material specific and depend on each particular filler, resin composition and ratio. Nevertheless, the effects of increased violet light transmittance through indirect materials are worthy of further investigation, especially for RBLCs containing photoinitiator chemistries that absorb as shorter wavelength bands.

The second obvious limitation to light curing RBLCs through thick layers was understanding the exact definition of “sufficient” transmittance. Perhaps a sensible approach would be a light transmittance high enough (for a given irradiation time, here 40 s) in order to reach a DC comparable to that obtained without any filter. Such threshold could be identified in the current work as ranging between 250 and 500 mW/cm^2 (Fig. 4). Such presentation of the data avoids arduous line-by-line analysis of the data tables (Tables 3–5), which often results in conclusions that are only relevant to each combination of filter types, shades, curing lights, etc. The ability to achieve an optimal DC purely by light curing depends on the combination of irradiance and irradiation time. It has been described before that there is “no apparent lower limit to the irradiance that may give effective polymerization, at least down to 25 mW/cm^2” [35]. This was reported for direct restorative composites, in thick layers (2 mm). In the context of RBLCs, where layers around 25 μm are used [31], this statement becomes even more relevant. As for the upper limit of irradiation time, it would be determined as the time a clinician is willing to devote to the light curing procedure, or risk of over-heating the pulp. Therefore, the question is not whether a light-curable RBLC can be cured optimally through thick indirect restorations, but rather, in what irradiation time (provided that a minimum irradiance reaches the material)? For the present work, this parameter was set at 40 s; other works (e.g. Ref. [36] indicated that maximum thickness for an efficient light cure during 20 s through ceramic filters is 2 mm. Therefore, it would be more appropriate to consider transmittance rather than filter material type, shade or thickness, and to adapt the irradiation time to provide specific indications for each luting material. Curing time exposure is the most critical parameter for optimizing degree of conversion.

Within the current curing parameters (40 s irradiation time), DC varied significantly with monomer ratio, photoinitiator type and curing light. Considering the monomer ratio, an increased low molecular weight monomers (TEGDMA) content led, as expected, to higher DC for RBLCs light cured under similar conditions [37,38]. This can be explained by their high molecular mobility, which enables additional propagation in the later stages of polymerization reaction, i.e. when it becomes diffusion-controlled [37,38]. Despite the attempt to formulate the experimental RBLCs in a comparable fashion to our control commercial material (Rely-X Veneer), it appeared that the optimal DC of the latter was in all cases inferior to the experimental formulations. Reasons for this may include the effect of proprietary compounds, pigments and other compounds that act as competitive absorbers, and/or a less favorable photoinitiator and co-monomer combination. Furthermore, although co-monomer ratio had an impact on the absolute DC values, it had no influence on the evolution of DC values with increasing filter thickness, which purely depends on light transmission.

Regarding the photoinitiator type, DC results confirmed previously described trends that for similar irradiance, resin composites or adhesives using Lu-TPO showed higher DC than their CQ counterparts (Fig. 4) [20,22,24,26,39,40]. Similarly, Lu-TPO-based light cured systems were associated first with a lower release of un-reacted monomers, hence with a lower cytotoxic potential [24], and second with superior mechanical properties compared to CQ-counterparts [23]. Higher mechanical properties may lead to a more effective, durable and stable bonding interface (tooth-RBLC and RBLC-indirect restorative material) over time with a reduced solubility [24], which remains to be verified.

The higher DC combined with higher mechanical properties of Lu-TPO-based composites were said to result in a higher cross-linking density, a characteristic which was also suggested to account for a higher color stability [20,41] and an improved resistance to hydrolytic degradation [20] compared with CQ-based materials. This further supports the potential of using Lu-TPO-based RBLC materials, to reduce the risk of interfacial degradation and discoloration. Such detrimental effects have been described in a greater proportion when using dual-cured materials compared with purely light cured types [9]. Other factors explaining the higher color stability may also relate to the oxidation process of the amine present in the CQ initiation system, which causes discolouration [21]. During photopolymerization, amines may also form by-products that can also cause yellow or brown discolouration [42]. The absence of amine in the catalyst system based on Lu-TPO could be associated with reduced shade alteration after aging. In summary, the use of Lu-TPO as photoinitiator in RBLCs has potential to improve clinical outcomes, both in terms of bonding efficiency, bonding stability as well as color stability. The latter is particularly important in case of thin veneers, since the final colour of these restorations after bonding to teeth were clearly shown to be influenced by the shade of the underlying structures, including the luting system [43].

Finally, the light spectrum and spectral irradiance has an important impact in terms of curing efficiency. Despite the increased transmitted irradiance of BPG2 (Fig. 3), which is probably due to the higher combined irradiance

of the two different spectrum present in the BPG2 light, DC values and profiles were similar for CQ-based materials, and higher for AURAviolet at lower irradiances (Fig. 4). This could be explained by the fact that while blue peaks between BPG2 and AURAblue were relatively comparable, the violet peak in BPG2 corresponds to only 20% of the total irradiance (Fig. 2). This results in a lower transmittance of the (~410 nm) violet spectrum at a given BPG2 transmittance value. Moreover, the emission peak of AURAviolet was located at shorter wavelengths (~405 nm), providing a more effective overlap with Lu-TPO absorption spectrum than the violet peak of the BPG2 (Fig. 2). For the commercial product Rely-X Veneer, the higher DC results associated with BPG2 compared with AURAblue at each level of transmittance was likely a result of the broader spectrum of the blue peak of the BPG2 compared to AURAblue, and consequently, a larger overlap with the absorption spectrum of CQ. Another less likely explanation would be the presence of an additional photoinitiating system in Rely-X Veneer absorbing at lower wavelengths, which would then benefit from the violet emission of the BPG2.

5 Significance

The current investigation confirmed that, under specific conditions, optimal photopolymerization of RBLCs could be achieved through indirect restorative materials (≤4 mm) and an irradiation time of 40 s. Such an approach, which is associated with both clinical advantages and fundamental improvements in material properties, may be viable, however, multiple factors such as monomer composition, photoinitiator content, filter material and thickness (studied here), and prolonged curing time (>-40 s) should be optimized accordingly.

The Lu-TPO-based RBLC provided higher conversion compared with the traditional CQ system, provided that sufficiently high irradiance in the violet wavelength range was used.

Finally, the performance of such an approach in terms of bond strength, bond stability, and ultimately clinical efficiency should be verified.

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Query: This opening parenthesis does not have a corresponding closing parenthesis. Please insert the parenthesis in the appropriate position.
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Query: Please provide the volume number and page range in Refs. [6,7].
Answer: Ref 6: The article was still in Press but now I can have the complete reference: J Prosthet Dent 2018, Volume 119, Issue 5, May 2018, Pages 743-748.e1

Query: Please check the edit made in Ref. [7], and correct if necessary.

Answer: Ok

Query: Please note that two sets of figure caption were provided. One in the manuscript and one in a separate file named "Figure captions.docx". We have used the one provided in the manuscript. Kindly check and correct if necessary.

Answer: The reference to the "B" is missing: please add "(B)" at the second line, after "irradiated with BluePhaseG2 (Ivoclar-Vivadent)"

Query: This closing parenthesis does not have a corresponding opening parenthesis. Please insert the parenthesis in the appropriate position.

Answer: Please remove the parenthesis after mW/cm2, and the corresponding parenthesis are "(" before mW/cm2 and ")" after 0,1

Query: Please check the presentation of all the Tables and correct if necessary.

Answer: The first table is incomplete; The other tables could take less space (see the document attached Tables.docx please), and there is a line not needed in the middle of the table 3

Attachments: Tables.docx