Mini-iFT Confirms Superior Adhesive Luting Performance using Light-curing Restorative Composites

Chloe M. F. Hardy[a] / Violette Landreau[b] / Margaux Valassis[c] / Ben Mercelis[d] / Jan De Munck[e] / Bart Van Meerbeek[f] / Julian Leprince[g]

Purpose: To validate the rationale of using a conventional light-curing resin-based composite (RBC) to lute thick indirect restorations by measuring mini-interfacial fracture toughness (mini-iFT).

Materials and Methods: Freshly exposed dentin of extracted third molars (n = 64) was immediately sealed with a thin layer of an experimental RBC with a 50 wt% or 75 wt% (IDS) filler load. Two- or 6-mm-thick CAD/CAM composite blocks were luted onto IDS using either pre-heated light-cure or dual-cure luting RBC, with the latter having served as control. Samples were cut into sticks, upon which a notch was prepared at the interface between IDS and luting RBC, prior to being submitted to a 4-point bending test to determine mini-iFT. The results were analyzed using a mixed linear model (LME). Failure mode at the fractured interface was determined using scanning electron microscopy (SEM).

Results: LME revealed that mini-iFT was not significantly affected by the composite block thickness ($p = 0.39$), but by the luting RBC ($p < 0.0001$) and the IDS RBC filler load ($p = 0.0011$). Mini-iFT was higher with 50 wt% filler-loaded RBC IDS and when luted using the light-curing RBC.

Conclusion: This work provides the proof of concept that 2- and 6-mm-thick indirect restorations can safely be adhesively luted with pre-heated conventional light-cure RBC under controlled light-irradiation conditions. This strategy even seems beneficial in terms of mini-iFT compared to using a dual-cure luting RBC. IDS with lower filler content also appeared more favorable.

Keywords: adhesion, composite resin, computer-aided design/computer-aided manufacturing (CAD/CAM), dental cement, dual cure, interfacial fracture toughness, light cure, resin-based luting composite.

J Adhes Dent 2021; 23: 539–548.
doi: 10.3290/j.jad.b2287755

Submitted for publication: 27.08.20; accepted for publication: 26.05.21

The results of this work were presented to the Academy of Dental Materials jury at the finals of the Paffenbarger's Award in 2019 during the Annual Meeting of the Academy of Dental Materials (October 2–5) at Jackson Hole, WY, USA.

Reconstruction of large defects in posterior teeth with indirect restorations is a conservative strategy with good clinical longevity.[3,6,60] Traditionally, those posterior indirect restorations are adhesively luted with luting resin-based composite (RBC) with a dual-cure setting system. This is to compensate for a potential lack of sufficient light-irradiance reaching the luting RBC through the indirect restorative material. However, the need of adequate light exposure of

[a] *Prosthodontist and PhD Student, School of Dental Medicine, Cliniques Universitaires Saint Luc, UCLouvain, Brussels, Belgium; DRIM Research Group, Advanced Drug Delivery and Biomaterials, Louvain Drug Research Institute (LDRI), UCLouvain, Brussels, Belgium. Idea, hypothesis, experimental design, performed the experiments in partial fulfillment of requirements for a degree, wrote the manuscript, performed statistical evaluation, contributed substantially to discussion.*

[b] *Prosthodontist, School of Dental Medicine, Cliniques Universitaires Saint Luc, UCLouvain, Brussels, Belgium. Performed the experiments in partial fulfillment of requirements for her specialization degree, assisted in conducting experiments.*

[c] *Prosthodontist, School of Dental Medicine, Cliniques Universitaires Saint Luc, UCLouvain, Brussels, Belgium. Performed the experiments in partial fulfillment of requirements for her specialization degree, assisted in conducting experiments.*

[d] *Lab Coordinator, KU Leuven (University of Leuven), Department of Oral Health Sciences, BIOMAT – Biomaterials Research group & UZ Leuven (University Hospitals Leuven), Dentistry, Leuven, Belgium. Coordination of the lab, assisted extensively with the research equipment.*

[e] *Dentist in Private Practice, former Researcher, KU Leuven (University of Leuven), Department of Oral Health Sciences, BIOMAT – Biomaterials Research group & UZ Leuven (University Hospitals Leuven), Dentistry, Leuven, Belgium. Development of the mini-IFT technique (experimental design), helped develop the experimental ideas.*

[f] *Professor, KU Leuven (University of Leuven), Department of Oral Health Sciences, BIOMAT – Biomaterials Research group & UZ Leuven (University Hospitals Leuven), Dentistry, Leuven, Belgium. Consulted on experimental design, contributed to the discussion, proofread the manuscript.*

[g] *Professor of Endodontics, School of Dental Medicine, Cliniques Universitaires Saint Luc, UCLouvain, Brussels, Belgium; DRIM Research Group, Advanced Drug Delivery and Biomaterials, Louvain Drug Research Institute (LDRI), UCLouvain, Brussels, Belgium. Idea, hypothesis, experimental design, assisted in statistical evaluation, contributed substantially to discussion, proofread the manuscript.*

Correspondence: *Chloe M. Hardy, Cliniques universitaires Saint Luc, UCLouvain, Avenue Hippocrate 10, 1200 Brussels, Belgium. UCLouvain, Brussels, Belgium. Tel: +32-49-962-1213; e-mail: chloe.hardy@uclouvain.be*

dual-cure luting RBC to reach optimal polymerization has been highlighted in numerous works.[1,16,19,23,36,41,52]

In addition, any dual-cure process requires a mix of two components, increasing the possibility of inhomogeneity and voids.[1] Beyond these considerations, several clinical advantages are associated with the use of purely light-cure luting RBC, notably a longer working time, shorter setting time and improved color stability.[26] These have led dental practitioners to use light-cure instead of dual-cure luting RBC to bond indirect composite or ceramic restorations. Such an approach has mostly been used so far for veneers in the anterior region, with good clinical success.[20,39] However, veneers are thin (typically < 1 mm) and usually quite translucent, unlike restorations in load-bearing areas, where weak light transmission is expected through thicker (> 2 mm) and usually less translucent restorations. As mentioned in a previous work, the obvious limitation is to achieve sufficient light transmittance for optimal polymerization of the luting RBC through the indirect restoration.[21] Given the inverse logarithmic relationship described between material thickness and light transmittance,[38] a very weak light irradiance is expected through thick layers (≥ 4 mm), typically below 250 mW/cm^2 or even below 50 mW/cm^2, depending on the specific combination of material (type, shade and thickness) and light characteristics.[22]

In terms of polymer chemistry, it has been stated that there is likely no lower irradiance limit to reach optimal polymerization, as long as sufficient irradiation times are provided.[38,55] Recently, it was confirmed that the optimal degree of conversion of luting RBC could be reached through a 4-mm block (zirconia or composite) using a 40-s irradiation time, but only in specific conditions (material type and shade, light wavelength, etc).[21] It was suggested that each specific condition requires the adjustment of irradiation time to reach optimal degree of conversion (DC), and that long irradiation times will likely be necessary in most instances. One simple option to increase polymerization efficiency is to preheat the RBC, thereby reducing at the same time composite viscosity and improving the flow.[1,9,47,57] This makes it possible to use conventional highly filled restorative RBC as luting RBC to bond indirect restorations.

The potential advantages of using high-modulus luting RBC have been highlighted in several in-vitro studies involving glass ceramics. It was shown that a higher elastic modulus and viscosity of the luting RBC resulted in increased film thickness,[53] ceramic strength,[8] and improved mechanical reliability.[8,53,54] Additionally, it was suggested that using a luting RBC with high elastic modulus and viscosity may positively influence the clinical longevity of resin-cemented glass-ceramic restorations.[2]

The available evidence supporting the use of light-cure restorative composites to lute thick indirect restorations is, however, limited. A few clinical studies compared the clinical performance of restorations placed with either dual- or light-cure luting RBC, but they were restricted to restorations with a maximum thickness of 2 mm, i.e. much thinner than extensive overlays or endocrowns, and mostly inlays, with the possibility for light to travel on the side of the restoration.[17,27,28] Looking into in vitro studies, a few investigated thick restorations, although they presented some limitations. Gregor et al[19] concluded that light-cure luting RBC could be cured through 7.5-mm-thick endocrowns, but this was based on an 80% microhardness ratio, without actually testing the interface. Moreover, the degree of "acceptable polymerization" has previously been described as being purely driven by convenience, without a rationale given for accepting sub-optimal cure,[38] thus possibly leading to sub-optimal material properties at depth.[32] Another study therefore considered the lack of statistical significance as the cut-off criterion, this time measuring DC through restorative materials up to 4 mm thick with a 40-s light-irradiation time.[21] While optimal cure could be achieved under certain conditions, the data again did not allow predicting interfacial quality. In contrast, Tomaselli et al[56] combined DC and microshear bond-strength measurements, but only up to 1.5-mm restoration thickness. To our knowledge, only Kameyama et al[25] compared the microtensile bond strength of thick restorations (8-mm-thick inlays) luted either with light-cure or dual-cure luting RBC, with promising results in favor of the light-cure material. However, the limitations were that adhesion was established on superficial dentin and that light was able to travel on the side of the restoration due to its small size relative to the light-tip diameter. Moreover, microtensile methods have since been challenged by an interesting approach, namely the mini-interfacial fracture toughness (mini-iFT),[43] which enables selectively testing the interface of interest. This makes it possible to test more complex scenarios with multiple interfaces, which is the case with the implementation of the so-called immediate dentin sealing (IDS) strategy. The latter consists of preparing the tooth for a bonded indirect restoration by immediately "sealing" the freshly cut dentin with adhesive and resin composite prior to impression. The main claimed advantages are improved bond strength, reduced dentin sensitivity[48,59] and blocked water uptake through osmosis from the underlying dentin (vital teeth).[34,61] While the need for systematic use of this technique has yet to be determined, a review of the literature reported that there is no scientific reason not to recommend it.[48] However, it may be seen as increasing the complexity of the procedure in the sense that a number of new variables may influence the outcome. Notably, the characteristics of the resin-based material (adhesive or composite) used to perform the IDS can vary, notably the filler load, which can potentially affect the quality of the interface with the luting material. It was recently reported that the use of adhesives with low or no filler content, such as for IDS, was associated with reduced bond strength.[10] Moreover, the surface treatment of the IDS technique prior to restoration placement is also different, since the IDS layer needs to be prepared differently from dental tissues, notably by sandblasting and application of coupling agents (silane and/or 10-methacryloyloxydecyl dihydrogen phosphate [10-MDP]). The latter is necessary in case the active chemical groups (free radicals and unconverted double bonds) are no longer available at the surface of the material, which was shown

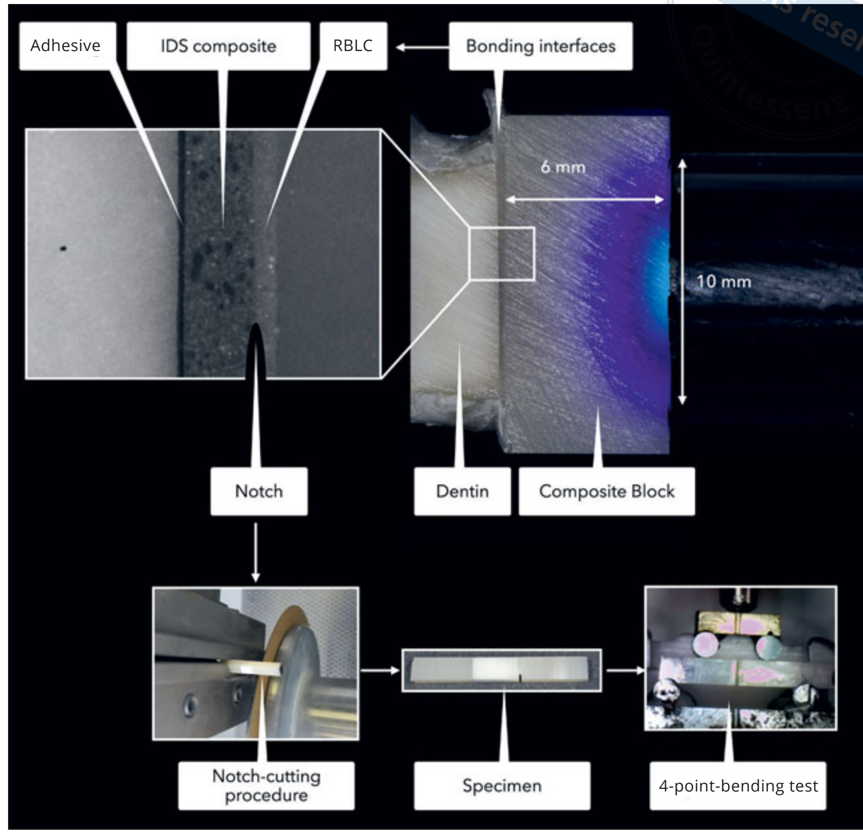

Fig 1 Illustration of specimen preparation procedure. The upper right picture shows the light tip of the Bluephase G2 (Ivoclar Vivadent) light curing unit next to a prepared tooth, showing how light attenuates as it goes through the composite block. The insert on the upper left shows the different interfaces under an optical microscope, and indicates the interface of interest where a notch was cut. The lower pictures illustrate notch preparation after the teeth were cut into bars, and mini-interfacial fracture toughness testing (mini-IFT) using an adapted 4-point bending test device. Note that in this example the luting composite was Panavia V5 (Kuraray Noritake). RBLC: resin-based luting composite.

to depend on time, material composition and storage conditions.[7,29-31] Optimal bond strength between the bonded indirect restoration and tooth with IDS was obtained when cementation was performed up to 12 weeks after IDS.[35]

In summary, using solely light-cure, highly filled RBC to lute thick indirect restorations, combined with the use of IDS, presents practical clinical advantages. However, the strategy requires more in vitro and clinical data to generalize its use. The goal of this work was to validate the rationale of this strategy by testing three null hypotheses with regard to mini-IFT: (1) block thickness (2 vs 6 mm) has no impact provided that DC is similar; (2) the use of light-curable composite is similar to dual-cure composite; and (3) the filler content (50 wt% vs 75 wt%) of the RBC used for IDS after cavity preparation has no impact.

MATERIAL AND METHODS

Sample Preparation

Sixty-four extracted human third molars were embedded in gypsum after the root was cut with a diamond disk at 950 rpm (Micracut 151, Kemet Metkon; Maidstone, Kent, UK) under constant water irrigation. The pulp chamber was emptied, cleaned with a diamond drill, and filled with the flowable RBC Clearfil Majesty Flow (Kuraray Noritake; Tokyo, Japan) after applying the 2-step self-etch adhesive Clearfil SE Bond 2 (C-SE2; Kuraray Noritake) following the manufacturer's instructions. After sandblasting (50-µm aluminum oxide, 2 bar, 1 cm from the surface, 5 s), the surface of a 7-mm-thick CAD/CAM composite block (Katana Avencia, shade A3; Kuraray Noritake) and preparing the tooth with Tooth Primer (Kuraray Noritake), the block was bonded to the filled tooth root using the luting RBC Panavia V5 (PV5; Kuraray Noritake). Immediately upon seating the block, the luting RBC was light cured from four sides for 40 s each (total: 160-s curing time) using the LED light-curing unit (LCU) Bluephase G2 (Ivoclar Vivadent; Schaan, Liechtenstein). The curing light was used in high power mode during the whole study, generating a light output of 1200 mW/cm^2, as confirmed by a Marc Resin Calibrator (BlueLight Analytics; Halifax, Canada). The bonded block yielded specimen lengths that enabled preparation of sufficiently long, symmetrical bars for mini-iFT testing. The interfaces of this specimen part were not tested. The present procedure was adapted based on previous work.[43]

The dental crown was then cut in the same way as the root to expose mid-coronal dentin without enamel, as verified using light microscopy (JSM-6610LV, JEOL; Tokyo, Japan). Next, a bonding procedure was immediately performed using C-SE2, followed by application of a thin layer of an experimentally prepared RBC, which corresponds to

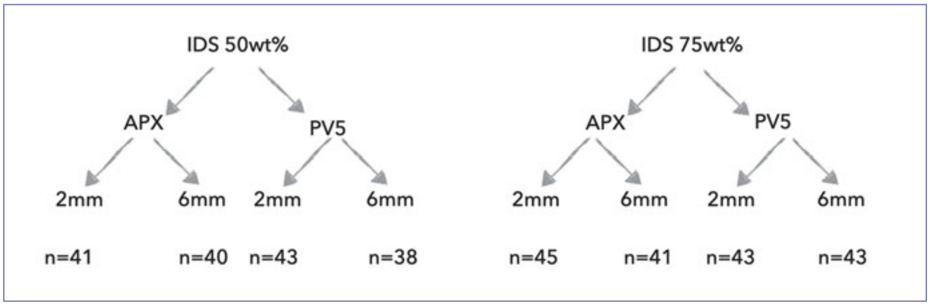

Fig 2 Summary of the different test groups. Eight third molars were prepared per condition. n = number of testable sticks for each group.

an immediate dentin sealing (IDS) procedure. For IDS, two experimental RBCs were prepared exactly as in previous work.[21] The RBCs were based on a TEG-DMA/bis-GMA mixture (50/50 wt%, Sigma-Aldrich; St Louis, MO, USA), to which a camphorquinone/amine (0.2/0.8 wt%, Sigma-Aldrich) photoinitiator system was added, with two different proportions of filler: 1. a filler load of 50 wt% (IDS_50 wt%), consisting of 40 wt% silanized barium-glass microfiller (G018-186/K6, Schott; Mainz, Germany: d50 = 3 ± 1 µm) and 10 wt% silanized fumed silica nanofiller (Aerosil R 7200, Evonik; Hanau, Germany: 12 nm); and 2. a filler load of 75 wt% (IDS_75 wt%), consisting of 65 wt% microfiller and 10 wt% nanofiller. The teeth prepared with IDS were then stored for one week at 37°C in distilled water, thus simulating a two-visit clinical procedure.

Luting Procedure

One week later, the IDS surface and the surface of the same CAD/CAM composite blocks (Katana Avencia; Kuraray Noritake) as used before were sandblasted (50-µm aluminum oxide, 2 bar, distance of 1 cm to surface, 5 s), copiously rinsed with water, and silanized (Clearfil Ceramic Primer Plus; Kuraray Noritake). The sandblasted blocks were next adhesively luted onto the sandblasted IDS-covered dentin, using either 1. the conventional light-cure RBC Clearfil AP-X (APX, Kuraray Noritake), which was preheated beforehand to 68°C using a composite heating device (Calset Tri-Tray Composite Heater, Kerr; Orange, CA, USA) after application and light curing (10 s) of C-SE2 following the manufacturer's instructions (test group), or 2. the dual-cure luting composite PV5 (control group). Both luting strategies involved light curing the luting RBC with the Bluephase G2 LCU solely from the top surface of the blocks (Fig 1), which were either 2 or 6 mm thick. The light-tip diameter was smaller than the block dimension to make sure that light could only reach the luting RBC through the block and was not able to travel along the block's side. The curing time was adjusted based on preliminary tests in order to reach non-significantly different degrees of conversion within the luting RBC, regardless of block thickness (2 or 6 mm, or without block). The resulting curing times required were 40 s and 240 s for the 2-mm- and 6-mm-thick blocks, respectively. The mean DC of APX was 71% (± 1.1), 76.6% (± 1.0), and 77.2% (± 2.9); the DC of PV5 was 55% (± 2.1), 52.1% (± 0.5) and 53.0% (± 2.6) ($p > 0.05$), respectively, without block interposition, or with 2- and 6-mm-thick blocks, as measured (n = 3) using a micro-Raman Spectrometer (DXR Raman Microscope, Thermo Scientific; Madison, WI, USA). The latter DC measurement procedure was performed as described previously.[21] The corresponding light irradiances received by the luting RBC, as measured with Thorlabs Optical Power and Energy Meter PM100USB (Thorlabs; Newton, NJ, USA) were the following: 1119 mW/cm^2 without block interposition, 98 mW/cm^2 through a 2-mm-thick block, and 4 mW/cm^2 through a 6-mm-thick block. A prepared specimen with the different interfaces involved is illustrated in Fig 1.

Mini-IFT Measurement

The prepared macrospecimens were stored for another week at 37°C in distilled water before being cut into sticks (microspecimens) of 1.5 x 2 mm (Accutom-50 with Cut-off Wheel M1D10, Struers; Ballerup, Denmark) under constant water irrigation. A notch was next cut on each stick (M1D08 diamond disk, Struers: 150 µm, cutting speed = 0.015 mm/s) exactly at the interface of interest, ie, between IDS and luting RBC (Fig 1). The notch was prepared under a stereomicroscope (Leica M715; Wetzlar, Germany). The acceptable range of the sticks and notch dimensions was based on the criteria established by Pongprueksa et al.[43] Eight teeth were prepared for each condition and all the samples that did not meet these criteria were discarded. The number of testable sticks per condition is given in Fig 2. The samples were submitted to a 4-point bending test (5848 Micro Tester, Instron; Norwood, MA, USA) at a cross-head speed of 0.05 mm/min (Fig 1). This provided the basis for calculating the mini-iFT.

Fracture Analysis

Following fracture, all specimens were prepared for SEM examination (JSM-6610LV, JEOL), as described by Pongprueksa et al.[43] The specimen dimensions were first measured using an optical microscope (400-NRC, Leitz; Oberkochen, Germany) at 250X magnification prior to SE microscopy to determine the origin of fracture and its propagation, as well as to search for potential flaws. Failure modes were classified as either adhesive, mixed, or cohesive.[50] Only adhesive failures were considered for calculation of mini-iFT.

Table 1 Mini-IFT results (MPa), standard deviation (SD), number of ptfs, percentage of interfacial fractures (%) and total number of testable sticks for each condition (n)

IDS composite	Thickness Katana Avencia Block	RBLC	ptf/n	Interfacial fractures (%)	Mean Mini-iFT (MPa $m^{1/2}$)	SD
50 wt%	2 mm	APX	0/41	94	1.46	0.53
50 wt%	2 mm	PV5	0/43	91	1.39	0.42
50 wt%	6 mm	APX	1/40	80	1.58	0.44
50 wt%	6 mm	PV5	0/38	100	1.1	0.44
75 wt%	2 mm	APX	0/45	94	1.33	0.37
75 wt%	2 mm	PV5	0/43	74	1.09	0.44
75 wt%	6 mm	APX	1/41	98	1.37	0.5
75 wt%	6 mm	PV5	1/43	86	1.05	0.5

ptfs: pre-test failures.

Statistical Analysis

Statistical analysis was performed using JMP Pro 14 software (SAS Institute JMP; Grégy-sur-Yerres, France). The normality of distributions was verified with the Shapiro-Wilk test. A mixed linear model was used, followed by Student's t-test. Tooth dependency[4] was taken into account using a random effects model.

RESULTS

SEM of the fractured surfaces revealed that most failures (74% to 100%) occurred at the interface between IDS and luting RBC (Table 1), ie, the interface of interest where the notch was cut (Figs 3b, 3c, 3f, 3h). A few specimens revealed mixed failure (Figs 3a and 3e), with the origin of fracture located at the interface but occasionally deviating into either luting RBC or IDS. A small number showed a fracture between the luting RBC and the composite block (Fig 3d). The number of pre-test failures (ptfs) was very low (Table 1). All samples included in the mini-iFT calculations exhibited either purely adhesive failure at the interface of interest, or mixed failure with the specimen tip and >50% of the total fractured surface having failed at the interface.

With regard to each null hypothesis, and considering the mini-iFT:

- There was no statistically significant difference between 2-mm- and 6-mm-thick blocks ($p = 0.3884$) (Table 2, Fig 4).
- The effect of luting RBC (APX and PV5) was highly statistically significant ($p < 0.0001$), where APX statistically significantly outperformed PV5 (Table 2, Fig 4).
- There was a statistically significant difference ($p < 0.0011$) between IDS_50 wt% and IDS_75 wt% (Table 2, Fig 4), where mini-iFT related to IDS_50 wt% was statistically significantly higher than that of IDS_75 wt%.
- The effect of the tooth as unit was not statistically significant ($p > 0.05$, random effects model).

DISCUSSION

The first null hypothesis, that block thickness (2 or 6 mm) had no impact on mini-iFT if the curing time was adapted to provide similar DC of the luting RBC, failed to be rejected ($p = 0.3884$). This study confirmed the conclusions of previous works,[19,25,56] that under certain conditions, conventional light-curing restorative composites can be used effectively as luting composites, thus filling the gaps left by those studies. All the light irradiated onto the luting RBC was transmitted through the indirect restorative CAD/CAM block and could not travel along its side to reach the luting RBC. The work also relies on what the authors believe is currently the most appropriate method to test the bonding quality of a specific interface, ie, the mini-iFT method. In this way, proof of concept is reached that indirect restorations up to a thickness of 6 mm can be luted using solely light-curable, highly filled restorative RBCs, but requires an extension of curing time to ensure optimal polymerization of the RBC. Our preliminary tests to determine the curing time required were essential, since minor changes in terms of DC can lead to major variance in mechanical properties, due to the RBC's resin phase changing states.[32] The importance of extending the curing time to optimize DC is supported by previous studies,[21,37,40] showing that even at very low light irradiance, optimal conversion of a conventional restorative RBC used as luting agent can be obtained, given a sufficiently long irradiation time. In the present study, an irradiance of 4 mW/cm^2 was measured through a 6-mm Katana Avencia (Kuraray Noritake) composite CAD/CAM block. Based on the preliminary tests, a curing time of 40 s and 240 s through a 2-mm- and a 6-mm-thick composite block, respectively, was required. This finding is also in line with

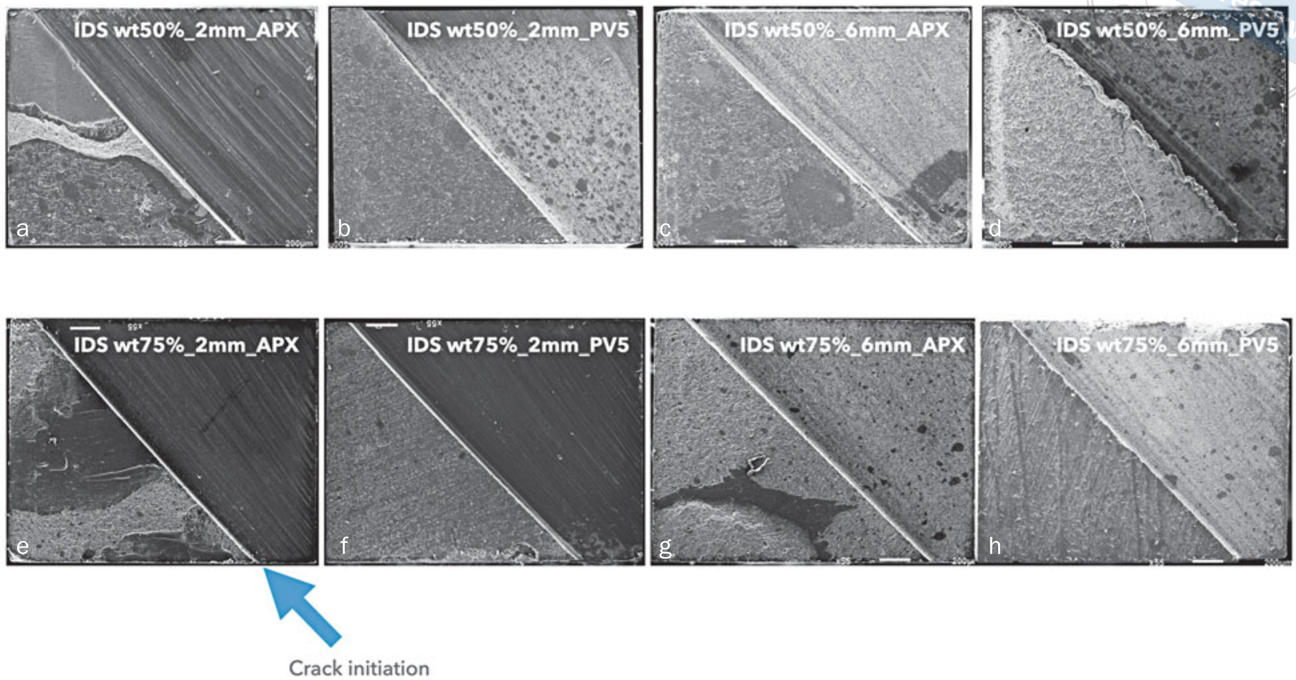

Fig 3 Representative SEM images of the fractured interfaces (one for each tested condition/type of IDS_block thickness_RBLC). a and e show mixed fractures with < 50% interfacial characteristics, so these samples were excluded; b, c, f, h show interfacial fractures, hence included; d shows an interfacial fracture, but between PV5 and the Katana block, so this sample was excluded; g depicts a mixed fracture with >50% interfacial characteristics, so this samples was included. The proportion of interfacial failures between the IDS composite and the RBLC were the following: 94% for IDS wt50%_2mm_APX, 91% for IDS wt50%_2mm_PV5, 80% for IDS wt50%_6mm_APX, 100% for IDS wt50%_6mm_PV5, 94% for for IDS wt75%_2mm_APX, 74% for IDS wt75%_2mm_PV5, 98% for IDS wt75%_6mm_APX and 86% for IDS wt75%_6mm_PV5.

more fundamental studies on dimethacrylate-based materials using a camphorquinone/amine photoinitiator system, for which long irradiation times at low irradiance have been described as more beneficial in terms of degree of conversion compared to the opposite, ie, short curing times at high irradiance.[13,42] The rationale has been explained as lower free-radical bimolecular termination with longer irradiation at lower irradiance, ultimately leading to an increased amount of polymer growth centers.[31] In this sense, two important aspects to consider are the LCU parameters and the loss of light as it travels through the indirect restorative material.

In terms of the objectives of the present work, the LCU must present a large curing-tip diameter to cover the restoration and provide homogeneous light distribution over the whole restoration surface. Moreover, such a homogeneous light beam should be maintained as much as possible when the distance from the light tip increases.[46] The LCU chosen for this study (Bluephase G2, Ivoclar Vivadent) fulfils these criteria quite well.[12,48,51] Only a slight light-beam inhomogeneity over the tip surface has been reported and attributed to the multiple LED chips (3 blue and one violet) present in the LCU, combined with a coherent light bundle guided towards the curing tip.[12] However, this slight light-beam inhomogeneity does not result in differences in degree of conversion or microhardness over the surface or with depth, at least not for materials containing a camphorquinone/amine-based photo-initiator system;[12,45] this must in part also be ascribed to light scattering within the composite.[12]

While light scattering within composite has a positive effect on polymerization homogeneity, it is at the same time the major factor responsible for light attenuation through the composite material, in addition to light absorption.[38,40] The inverse logarithmic relationship described between composite material thickness and light transmittance[21,38] was again verified in this work (data not shown). As reported before, the curve slope is specific to each curing light/material combination,[21,22] and the values reported in the present work are therefore only valid for the exact combination of parameters set in this study. Specifically in terms of light curing through indirect RBC restorations, such as those in this study, the factors influencing the material thickness/light transmittance curve slope are expected to be mostly related to light scattering by composite filler particles (hence the mismatch between filler type, size and load, as well as resin-filler refractive index), and to light absorption by pigments (hence material shade).[14] The composite block used here (Katana Avencia, Kuraray Noritake) is exclusively filled with nanofiller particles (20 and 40 nm, 62 wt% filler load) infused with resin monomers before being polymerized under high pressure and temperature.[58] Concerning the optical effects of filler-particle properties on light transmittance, light scattering is considered maximum if the filler size is about half the wavelength of the light.[14] This is not the case here, which should be in favor of light transmission through the RBC block.[18] This means that light transmission through Katana Avencia (Kuraray Noritake) blocks is weaker than through unfilled resin, but should be higher than through composite blocks containing high proportions of larger filler particles.

The second null hypothesis was rejected, since significant differences in mini-iFT were observed between the two luting RBCs tested ($p < 0.0001$), favoring the light-curing composite APX. Previous research comparing adhesive luting of indirect restorations with either light- or dual-curing RBCs is limited. The few clinical studies that investigated the matter in posterior restorations showed a minor difference between the two luting strategies.[27,28] One 12-year clinical study showed significantly more bulk fracture when ceramic inlays and onlays were luted with a conventional RBC,[17] although clinical application advantages, such as excess removal, were clearly mentioned in favor of using high-viscosity light-curing RBCs as luting agents. However, although those studies involved only relatively thin inlays and onlays (< 2 mm), concerns were raised regarding possibly insufficient polymerization of the light-curing materials. To our knowledge, only two studies have investigated this question regarding thicker indirect restorations (>4 mm). One measured microhardness of the luting RBC under the indirect restoration, which is not a property that enables direct comparison of bonding quality obtained with light- vs dual-curing polymerization strategies.[19] In contrast, the other study[25] measured microtensile bond strength of 8-mm-thick ceramic inlays to dentin, and reported lower bond strength using the dual-curing luting RBC than using the light-curing RBC.[25] Despite being promising, these results required confirmation, since 8-mm-thick inlays are rarely placed in occlusal cavities, and more importantly, the study design allowed light travelling along the side of the inlay, which is not clinically realistic for a large overlay or endocrown, where light can only go through the bulk of the material. However, the present study, which used the same RBC APX as in Kameyama et al,[25] confirmed a similar trend, showing improved bonding with the high-viscosity restorative RBC. However, it must be mentioned that in this study, the light-curing RBC was preheated at 68°C, to initially increase the flowability of the material for luting purposes.

Pre-heating composite prior to photo-activation was shown to provide greater degrees of conversion, accelerate polymerization, and reduce curing time.[1,9,47,57] Restoration seating and removing RBC excess are clinically easier with (pre-heated) conventional paste-like than flowable RBCs, with the latter including dual-cure luting composites. Another clinical advantage is that multiple indirect restorations in one quadrant can be luted simultaneously using a light-curing RBC, as unlimited seating/luting time is available. It was also reported that an RBC light cured through a composite onlay (Signum, Heraeus Kulzer; Hanau, Germany) of 2-, 3- or 4-mm thickness presented a significantly higher DC (up to 30% for 4-mm-thick restorations) when previously preheated at 54°C, as compared to ambient temperature.[1] Our preliminary data, obtained by micro-Raman spectrometry conducted under the conditions of this study, confirmed a similar trend for APX (data not shown), so that pre-heating was implemented in the light-curing RBC test group in this study, as it was advantageous in several ways.

Table 2 Influence of the different parameters on the mini-IFT: p-values (mixed linear model)

3-way ANOVA	Prob. > |t|
Intercept	<0.0001*
wt% fillers IDS	0.0011*
Block thickness	0.3884
RBLC	<.0001*
wt% fillers IDS * block thickness	0.4423
wt% filler IDS * RBLC	0.8898
Block thickness * RBLC	0.0222*

The third null hypothesis was rejected as well, since the IDS filler content significantly affected mini-iFT ($p = 0.0011$), with higher mini-iFT recorded for the IDS RBC with lower filler content (50 wt%). This finding points out that the IDS filler content has an impact on the quality of the adhesive interface. This result tends to show that the resin phase may contribute more to the mini-iFT than the filler, despite appropriate surface conditioning conducted. When implementing IDS, it has indeed been recommended to sandblast the outer IDS layer, with IDS usually involving only the bonding resin or with additional RBC applied. Sandblasting removes any contamination by the impression material, bacteria, plaque, or provisional material, which could potentially hamper bonding of the luting RBC applied on top during the restoration-placement procedure.[15] In addition, sandblasting not only microroughens the IDS surface, promoting micromechanical interlocking, but also exposes the filler particles, making them available for surface conditioning and increasing their ability to react with the methacrylate groups of the RBC employed for luting. Depending on the filler nature, silane and/or 10-MDP are recommended to be applied on IDS composite; a combined 10-MDP/silane ceramic primer (Clearfil Ceramic Primer Plus, Kuraray Noritake) was used in this study. However, the resin phase

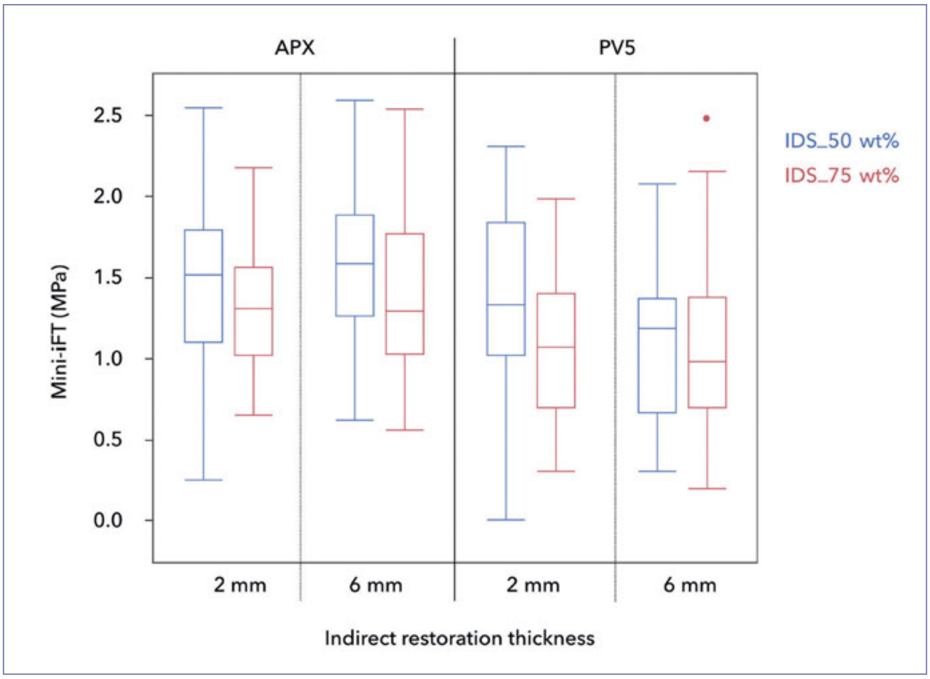

Fig 4 Box plots of mini-IFT data. Values corresponding to APX are on the left, and values for PV5 are on the right, each of them either with 2- or 6-mm-thick blocks. IDS 50 wt% values are in blue and IDS 75 wt% in red.

at the IDS surface still contains chemically active species, more specifically, unconverted methacrylate groups and trapped free radicals.[33] Given the results of the present work, these species may have contributed more to enhanced interfacial bonding of the luting RBC to the IDS RBC than did exposed filler surfaces.

Trapped free radicals can be detected in dental resin-based materials, eg, RBCs, for weeks or even months after light curing.[7,30,31] Their half-life depends on temperature, filler fraction, type and surface conditioning, and storage medium.[7,30] Unconverted double bonds were also detected at the surface of dental resins for weeks after polymerization, even when stored in water or artificial saliva; ethanol, however, led to a more rapid disappearance of reactive methacrylate groups at the surface.[29] The persistence of these active species is in line with the data reported by Magne et al,[35] showing that optimal bond strength of the indirect restoration bonded to the tooth via IDS can be achieved several weeks after the IDS procedure. The persistence of trapped free radicals and unconverted double bonds at the surface of the IDS composite probably accounts for the superior mini-iFT measured in the present work when using IDS_50 wt%, which has the highest resin content, in which more chemically active groups remained. If the luting composite primarily bonded to the IDS composite via silane treatment and connection to the exposed filler particles, the outcome would show a higher mini-iFT for the IDS composite filled at 75 wt%. The usefulness of silanization before adhesive luting a week after IDS completion is therefore to be questioned. A recent prospective randomized clinical trial on the subject showed that a recently placed IDS composite (two weeks under temporary filling) can be effectively cleaned by pumice rubbing only, without producing significant differences in microtensile bond strength as compared to tribochemical silica coating, while the latter led to significantly higher bond strength after 6-month storage.[59] Again, this finding is likely the result of the decrease over time of the chemically active species within the IDS composite. It is worth noting that the instructions for PV5 do not mention using it in association with IDS. It is advised to apply 'Tooth Primer' (Kuraray Noritake) on the dental substrate, which was not done here due to the different nature of the IDS substrate. Pilot experiments – under conditions similar to those used for the mini-IFT measurements – were performed to identify a possible impact of applying Tooth Primer (Kuraray Noritake) on the degree of conversion of PV5; no significant differences in DC ($p > 0.05$) were noticed with and without Tooth Primer (Kuraray Noritake) applied on the IDS. The conclusions of the present study are only valid for a clinical strategy in which IDS is used, and the work will need to be repeated for clinicians luting directly on tooth substrate.

Finally, the choice of mini-iFT to characterize the quality of the interface can be discussed. In this past, this method was shown to provide equivalent results to the well-documented microtensile bond strength tests.[43] Potential advantages of interfacial fracture toughness approaches over bond strength methods were reported by several groups, the former being described as more reliable, with failures observed at the interface of choice, with less variation.[11,24,43] However, most papers on the subject also reported increased methodological complexity,[11,24,43] which can be discouraging for researchers.[44] In the context of the present investigation, where multiple interfaces are present (Fig 1), the use of an interfacial method such as mini-iFT seemed the only appropriate approach, since it enabled specifically selecting the interface that was considered important to test. Scanning electron microscopy of the fractured surfaces confirmed the ability of the method to induce crack propagation where needed. While there has been some criticism in the past regarding the method,[5,49] there was to our knowledge no better alternative to test the present hypotheses, although the absolute mini-iFT values remain to be further validated.

CONCLUSION

The present work provides proof of concept that 1. adhesive luting with conventional light-curing restorative RBC is a good option for luting indirect composites, 2. the thickness of the restoration is not a problem if the curing time is adapted to the thickness and nature of the composite block, and 3. IDS with regard to particle-filler load has an impact on its bonding receptiveness. Of course, the ultimate validation through clinical studies is required.

Although the findings of the present work cannot be directly extrapolated to other conditions (other indirect material types, shades, light-curing units, etc), they provide guidance to optimize the procedure for these other conditions, in order to ensure effective luting when using solely light-curing composites. Given the difficulty of mini-iFT measurements and based on the present proof of concept, one option would be to optimize curing parameters by measuring the DC of the RBC and adjust the conditions to reach optimal DC.

ACKNOWLEDGMENTS

C.M.F.H and J.G.L. would like to thank the Cliniques Universitaire St Luc for their FRC funding. The authors are grateful to the manufacturers for suppling the materials (Kuraray Noritake, Ivoclar Vivadent, AdDent), and to Lieven Desmet (SMCS UCLouvain) for his help with the statistical analysis. The authors also thank Kuraray Noritake Europe for covering some of the operating costs of this study.

REFERENCES

1. Acquaviva PA, Cerutti F, Adami G, Gagliani M, Ferrari M, Gherlone E, Cerutti A. Degree of conversion of three composite materials employed in the adhesive cementation of indirect restorations: a micro-Raman analysis. J Dent 2009;37:610–615.
2. Addison O, Sodhi A, Fleming GJ. Seating load parameters impact on dental ceramic reinforcement conferred by cementation with resin-cements. Dent Mater 2010;26:915–921.
3. Archibald JJ, Santos GC Jr, Moraes Coelho Santos MJ. Retrospective clinical evaluation of ceramic onlays placed by dental students. J Prosthet Dent 2018;119:743–748.e1
4. Armstrong S, Breschi L, Ozcan M, Pfefferkorn F, Ferrari M, Van Meerbeek B. Academy of Dental Materials guidance on in vitro testing of dental composite bonding effectiveness to dentin/enamel using micro-tensile bond strength (muTBS) approach. Dent Mater 2017;33:133–143.
5. Belli R, Zorzin JI, Lohbauer U. Fracture toughness testing of dental restoratives: a critical evaluation. Curr Oral Health Rep 2018;5:163–168.
6. Blatz MB, Vonderheide M, Conejo J. The effect of resin bonding on long-term success of high-strength ceramics. J Dent Res 2018;97:132–139.
7. Burtscher P. Stability of radicals in cured composite materials. Dent Mater 1993;9:218–221.
8. Coelho NF, Barbon FJ, Machado RG, Boscato N, Moraes RR. Response of composite resins to preheating and the resulting strengthening of luted feldspar ceramic. Dent Mater 2019;35:1430–1438.
9. Daronch M, Rueggeberg FA, De Goes MF. Monomer conversion of pre-heated composite. J Dent Res 2005;84:663–667.
10. de Carvalho MA, Lazari-Carvalho PC, Polonial IF, de Souza JB, Magne P. Significance of immediate dentin sealing and flowable resin coating reinforcement for unfilled/lightly filled adhesive systems. Journal of esthetic and restorative dentistry : J Esthet Restor Dent 2021;33:88–98.
11. De Munck J, Luehrs AK, Poitevin A, Van Ende A, Van Meerbeek B. Fracture toughness versus micro-tensile bond strength testing of adhesive-dentin interfaces. Dent Mater 2013;29:635–644.
12. de Oliveira D, Rocha MG, Correr AB, Ferracane JL, Sinhoreti M. Effect of beam profiles from different light emission tip types of multiwave light-emitting diodes on the curing profile of resin-based composites. Oper Dent 2019;44:365–378.
13. Dewaele M, Asmussen E, Peutzfeldt A, Munksgaard EC, Benetti AR, Finne G, Leloup G, Devaux J. Influence of curing protocol on selected properties of light-curing polymers: degree of conversion, volume contraction, elastic modulus, and glass transition temperature. Dent Mater 2009;25:1576–1584.
14. dos Santos GB, Alto RV, Filho HR, da Silva EM, Fellows CE. Light transmission on dental resin composites. Dent Mater 2008;24:571–576.
15. Falkensammer F, Arnetzl GV, Wildburger A, Krall C, Freudenthaler J. Influence of different conditioning methods on immediate and delayed dentin sealing. J Prosthet Dent 2014;112:204–210.
16. Flury S, Peutzfeldt A, Lussi A. The effect of polymerization procedure on Vickers hardness of dual-curing resin cements. Am J Dent 2011;24:226–232.
17. Frankenberger R, Taschner M, Garcia-Godoy F, Petschelt A, Kramer N. Leucite-reinforced glass ceramic inlays and onlays after 12 years. J Adhes Dent 2008;10:393–398.
18. Fujita K, Ikemi T, Nishiyama N. Effects of particle size of silica filler on polymerization conversion in a light-curing resin composite. Dent Mater 2011;27:1079–1085.
19. Gregor L, Bouillaguet S, Onisor I, Ardu S, Krejci I, Rocca GT. Microhardness of light- and dual-polymerizable luting resins polymerized through 7.5-mm-thick endocrowns. J Prosthet Dent 2014;112:942–948.
20. Gresnigt MM, Kalk W, Ozcan M. Clinical longevity of ceramic laminate veneers bonded to teeth with and without existing composite restorations up to 40 months. Clin Oral Investig 2013;17:823–832.
21. Hardy CMF, Bebelman S, Leloup G, Hadis MA, Palin WM, Leprince JG. Investigating the limits of resin-based luting composite photopolymerization through various thicknesses of indirect restorative materials. Dent Mater 2018;34:1278–1288.
22. Ilie N. Transmitted irradiance through ceramics: effect on the mechanical properties of a luting resin cement. Clin Oral Investig 2017;21:1183–1190.
23. Ilie N, Hickel R. Correlation between ceramics translucency and polymerization efficiency through ceramics. Dent Mater 2008;24:908–914.
24. Ilie N, Ruse ND. Shear bond strength vs interfacial fracture toughness – Adherence to CAD/CAM blocks. Dent Mater 2019;35:1769–1775.
25. Kameyama A, Bonroy K, Elsen C, Luhrs AK, Suyama Y, Peumans M, Van Meerbeek B, De Munck J. Luting of CAD/CAM ceramic inlays: direct composite versus dual-cure luting cement. Biomed Mater Eng 2015;25:279–288.
26. Kilinc E, Antonson SA, Hardigan PC, Kesercioglu A. Resin cement color stability and its influence on the final shade of all-ceramics. J Dent 2011;39(suppl 1):e30–36.
27. Kramer N, Frankenberger R. Clinical performance of bonded leucite-reinforced glass ceramic inlays and onlays after eight years. Dent Mater 2005;21:262–271.
28. Kramer N, Reinelt C, Richter G, Frankenberger R. Four-year clinical performance and marginal analysis of pressed glass ceramic inlays luted with ormocer restorative vs. conventional luting composite. J Dent 2009;37:813–819.
29. Lee SY, Greener EH, Mueller HJ. Effect of food and oral simulating fluids on structure of adhesive composite systems. J Dent 1995;23:27–35.
30. Leprince J, Lamblin G, Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Mestdagh M, Leloup G. Kinetic study of free radicals trapped in dental resins stored in different environments. Acta Biomater 2009;5:2518–2524.
31. Leprince JG, Lamblin G, Devaux J, Dewaele M, Mestdagh M, Palin WM, Gallez B, Leloup G. Irradiation modes' impact on radical entrapment in photoactive resins. J Dent Res 2010;89:1494–1498.

32. Leprince JG, Leveque P, Nysten B, Gallez B, Devaux J, Leloup G. New insight into the "depth of cure" of dimethacrylate-based dental composites. Dent Mater 2012;28:512–520.
33. Leprince JG, Palin WM, Hadis MA, Devaux J, Leloup G. Progress in dimethacrylate-based dental composite technology and curing efficiency. Dent Mater 2013;29:139–156.
34. Luhrs AK, De Munck J, Geurtsen W, Van Meerbeek B. Composite cements benefit from light-curing. Dent Mater 2014;30:292–301.
35. Magne P, So WS, Cascione D. Immediate dentin sealing supports delayed restoration placement. J Prosthet Dent 2007;98:166–174.
36. Meng X, Yoshida K, Atsuta M. Influence of ceramic thickness on mechanical properties and polymer structure of dual-cured resin luting agents. Dent Mater 2008;24:594–599.
37. Musanje L, Darvell BW. Polymerization of resin composite restorative materials: exposure reciprocity. Dent Mater 2003;19:531–541.
38. Musanje L, Darvell BW. Curing-light attenuation in filled-resin restorative materials. Dent Mater 2006;22:804–817.
39. Nejatidanesh F, Savabi G, Amjadi M, Abbasi M, Savabi O. Five year clinical outcomes and survival of chairside CAD/CAM ceramic laminate veneers - a retrospective study. J Prosthodont Res 2018;62:462–467.
40. Palin WM, Leprince JG, Hadis MA. Shining a light on high volume photocurable materials. Dent Mater 2018;34:695–710.
41. Passos SP, Kimpara ET, Bottino MA, Santos GC, Jr., Rizkalla AS. Effect of ceramic shade on the degree of conversion of a dual-cure resin cement analyzed by FTIR. Dent Mater 2013;29:317–323.
42. Peutzfeldt A, Asmussen E. Resin composite properties and energy density of light cure. J Dent Res 2005;84:659–662.
43. Pongprueksa P, De Munck J, Karunratanakul K, Barreto BC, Van Ende A, Senawongse P, Van Meerbeek B. Dentin Bonding Testing Using a Mini-interfacial Fracture Toughness Approach. J Dent Res 2016;95:327–333.
44. Pongprueksa P, De Munck J, Karunratanakul K, Barreto BC, Van Ende A, Senawongse P, Van Meerbeek B. Response to Letter to the Editor, "Dentin Bonding Testing Using a Mini-interfacial Fracture Toughness Approach". J Dent Res 2016;95:954.
45. Price RB, Labrie D, Rueggeberg FA, Sullivan B, Kostylev I, Fahey J. Correlation between the beam profile from a curing light and the microhardness of four resins. Dent Mater 2014;30:1345–1357.
46. Price RB, Rueggeberg FA, Labrie D, Felix CM. Irradiance uniformity and distribution from dental light curing units. J Esthet Restor Dent 2010;22: 86–101.
47. Price RB, Whalen JM, Price TB, Felix CM, Fahey J. The effect of specimen temperature on the polymerization of a resin-composite. Dent Mater 2011; 27:983–989.
48. Qanungo A, Aras MA, Chitre V, Mysore A, Amin B, Daswani SR. Immediate dentin sealing for indirect bonded restorations. J Prosthodont Res 2016;60:240–249.
49. Ruse ND. Letter to the Editor, "Dentin bonding testing using a mini-interfacial fracture toughness approach". J Dent Res 2016;95:953.
50. Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. Dent Mater 2010;26:e78–93.
51. Shimokawa CA, Turbino ML, Harlow JE, Price HL, Price RB. Light output from six battery operated dental curing lights. Mater Sci Eng C Mater Biol Appl 2016;69:1036–1042.
52. Soares CJ, da Silva NR, Fonseca RB. Influence of the feldspathic ceramic thickness and shade on the microhardness of dual resin cement. Oper Dent 2006;31:384–389.
53. Spazzin AO, Bacchi A, Alessandretti R, Santos MB, Basso GR, Griggs J, Griggs J, Morale RR. Ceramic strengthening by tuning the elastic moduli of resin-based luting agents. Dent Mater 2017;33:358–366.
54. Spazzin AO, Guarda GB, Oliveira-Ogliari A, Leal FB, Correr-Sobrinho L, Moraes RR. Strengthening of porcelain provided by resin cements and flowable composites. Oper Dent 2016;41:179–188.
55. Stawarczyk B, Awad D, Ilie N. Blue-light transmittance of esthetic monolithic CAD/CAM materials with respect to their composition, thickness, and curing conditions. Oper Dent 2016;41:531–540.
56. Tomaselli LO, Oliveira D, Favarao J, Silva AFD, Pires-de-Souza FCP, Geraldeli S, Coelho Sinhoreti MA. Influence of pre-heating regular resin composites and flowable composites on luting ceramic veneers with different thicknesses. Braz Dent J 2019;30:459–466.
57. Trujillo M, Newman SM, Stansbury JW. Use of near-IR to monitor the influence of external heating on dental composite photopolymerization. Dent Mater 2004;20:766–777.
58. Tsujimoto A, Barkmeier WW, Takamizawa T, Latta MA, Miyazaki M. Influence of thermal cycling on flexural properties and simulated wear of computer-aided design/computer-aided manufacturing resin composites. Oper Dent 2017;42:101–110.
59. van den Breemer C, Ozcan M, Cune MS, Ayres AA, Van Meerbeek B, Gresnigt M. Effect of immediate dentin sealing and surface conditioning on the microtensile bond strength of resin-based composite to dentin. Oper Dent 2019;44:E289–E298.
60. van Dijken JW, Hasselrot L. A prospective 15-year evaluation of extensive dentin-enamel-bonded pressed ceramic coverages. Dent Mater 2010;26: 929–939.
61. Van Landuyt KL, Snauwaert J, De Munck J, Coutinho E, Poitevin A, Yoshida Y, Suzuki K, Lambrechts P, Van Meerbeek B. Origin of interfacial droplets with one-step adhesives. J Dent Res 2007;86:739–744.

Clinical relevance: The use of a conventional light-cure resin-based composite to lute thick indirect restorations (up to 6 mm) is a rational strategy, both in terms of its clinical advantages and the improved mini-interfacial fracture toughness. However, this requires careful selection of materials and irradiation parameters.