MINI-IFT CONFIRMS SUPERIOR ADHESIVE LUTING PERFORMANCE USING LIGHT-CURE RESTORATIVE COMPOSITES

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Abstract:

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

MATHERIAL AND METHODS: Freshly exposed dentin of extracted third molars (n=64) was immediately sealed with a thin layer of an experimental RBC filled at 50 or 75wt% (IDS). A 2- and 6-mm thick CAD/CAM composite block was luted onto IDS using either pre-heated light-cure or dual-cure luting RBC 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 4-point bending 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 mini-iFT was not significantly affected by the composite block thickness ($p=0.39$) but by luting RBC ($p<0.0001$) and the IDS RBC filler load ($p=0.0011$). Mini-iFT was higher with 50wt% filler-loaded RBC IDS and when luted using the light-cure 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 seems even beneficial in terms of mini-iFT compared to using a dual-cure luting RBC. IDS with lower filler content appeared also more favourable.

Introduction

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 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 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 interest of using high-modulus luting RBC has been highlighted in several *in-vitro* studies involving glass ceramics. It was indeed shown that a higher elastic modulus and viscosity of the luting RBC resulted in increased film thickness (53), ceramic strengthening (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 maximum 2-mm thick restorations, 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* literature, a few studies investigated the question for 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 it was based on an 80% microhardness ratio, hence without actually testing the interface. Moreover, this degree of "acceptable polymerization" has previously been described as being purely driven by convenience, without a rationale given for accepting sub-optimal cure (38), hereby possibly leading to sub-optimal material properties at depth (32). Another work has therefore considered the lack of statistical significance as cut-off value, this time measuring DC through restorative materials up to 4 mm with 40-s light-irradiation time (21). While optimal cure could be achieved under specific conditions, the data again did not allow to predict interfacial quality. On the contrary, Tomaselli et al. (56) combined DC and micro-shear bond-strength measurements, but only up to 1.5-mm restoration thickness. To our knowledge, only Kameyama et al. (25) compared the micro-tensile 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 the adhesion was made on superficial dentin and that light was able to travel on the side of the restoration due to the small size of the restoration relative to the light tip diameter. Moreover, micro-tensile methods have since been challenged by an interesting approach, namely the mini-interfacial fracture toughness (mini-iFT) (43), which enables to test selectively 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 to improve bond strength, reduce dentin sensitivity (48, 59) and block 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 a complexification 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 as IDS was associated with reduced bond strength (10). Moreover, the surface treatment of the IDS prior to restoration placement is also different since the IDS layer indeed 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 to depend on time, material composition and storage conditions (7, 29-31). Optimal bond strength between the indirect bonded restoration and tooth with IDS was obtained when cementation was performed up to 12 weeks after the IDS (35).

In summary, using solely light-cure highly filled RBC to lute thick indirect restorations, combined with the use of IDS, present clinical practical 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 vs. 75wt%) 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 disc at 950 rpm (Micracut 151, Metkon, Kemet, 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. Upon having sandblasted (50-µm aluminium oxyde, 2 bar, distance of 1 cm to surface, 5 sec) the surface of a 7-mm thick CAD/CAM composite block (Katana Avencia, shade A3; Kuraray Noritake) and prepared the tooth with Tooth Primer (Kuraray Noritake), the block was bonded to the root part of the tooth (with the pulp chamber beforehand filled with flowable composite) 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 (totaling to 160-s curing time) using the LED light-curing unit (LCU) Bluephase G2 (Ivoclar Vivadent, Schaan, Liechtenstein), which was used in high power mode in 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 generated sufficient specimen length to be able to prepare sufficiently long and symmetrical bars for mini-iFT testing; the involved 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 part, now exposing mid-coronal dentin with absence of enamel, as having been verified using light microscopy (JSM-6610LV; JEOL, Tokyo, Japan). Next, a bonding procedure was immediately performed using C-SE2, followed by the application of a thin layer of an experimentally prepared RBC in light of an immediate dentin sealing (IDS) procedure. For IDS, two experimental RBCs were prepared exactly as done in previous work (21). The RBCs were based on a TEGDMA/BisGMA mixture (50/50wt%; Sigma-Aldrich, Saint Louis, MO, USA), to which a camphorquinone/amine (0.2/0.8wt%; Sigma-Aldrich) photoinitiator system was added and two different proportions of filler: one with a filler load of 50wt% (being referred to as 'IDS_50wt%'), consisting of 40wt% silanized barium-glass microfiller (G018-186/K6; Schott, Mainz, Germany: d50 = 3±1 µm) and 10wt% silanized fumed silica nanofiller (Aerosil R 7200; Evonik, Hanau-Wolfgang, Germany: 12 nm), and the second one filled at 75wt% ('IDS_75wt%'), consisting of 65wt% microfiller and 10wt% nanofiller. The prepared teeth with IDS were next stored during one week at 37°C in distilled water, hereby 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 aluminium oxyde, 2 bar, distance of 1 cm to surface, 5 sec), abundantly 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 beforehand preheated to 68°C using a composite-heating device (Calset Tri-Tray Composite Heater; Kerr, Orange, CA, USA), after application and light-curing (10 sec) of C-SE2 following the manufacturer's instructions (= test group), or (2) the dual-cure luting composite PV5 (= control group). Both luting strategies involved luting RBC light curing with Bluephase G2 from solely the top surface of the block (Figure 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 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 required curing times were 40 s and 240 s for, respectively, the 2-mm and 6-mm thick blocks. Mean DC of APX was 71% (±1.1), 76.6% (±1.0) and 77.2% (±2.9), and DC of PV5 55% (±2.1), 52.1% (±0.5) and 53.0% (±2.6) ($p>0.05$), respectively, without any block interposition, 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 Figure 1.

Mini-IFT measurement

The prepared macro-specimens were stored for another week at 37°C in distilled water, before being cut into sticks (micro-specimens) of 1.5 by 2 mm (Accutom-50 with Cut-off Wheel M1D10, Struers, Denmark) under constant water irrigation. A notch was next cut on each stick (M1DO8 diamond disk, Struers, Ballerup, Denmark: 150 µm, cutting speed = 0.015 mm/s) exactly at the interface of interest, i.e. between IDS and luting RBC (Figure 1). The notch was prepared under a stereo microscope (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 are mentioned in Figure 2. The samples were submitted to a 4-point bending test (5848 Micro Tester, Instron, Norwood, MA, USA: cross-head speed = 0.05 mm/min) (Figure 1), based on which the mini-iFT was calculated.

Fracture analysis

Following fracture, all specimens were prepared for examination using scanning electron microscopy (SEM; JSM-6610LV; JEOL), as described before by Pongprueksa et al. (43). The specimen dimensions were first measured using an optical microscope (400-NRC, Leitz, Oberkochen, Germany) at 250× magnification, prior to being examined using SEM to determine the origin of fracture and its propagation, as well as to search for potential imperfections. The failure mode was classified as either 'interfacial', 'mixed', or 'cohesive' failure (50); only adhesive (interfacial) failures were considered for calculation of mini-iFT.

Statistical analysis

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

Results

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

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

- There was no significant difference between 2-mm and 6-mm thick blocks ($p=0.3884$) (Table 2, Figure 4).
- The effect of luting RBC (APX and PV5) was highly significant ($p<0.0001$), APX significantly outperforming PV5 (Table 2, Figure 4).
- There was a significant difference ($p<0.0011$) between IDS_50wt% and IDS_75wt% (Table 2, Figure 4), mini-iFT related to IDS_50wt% being significantly higher than that of IDS_75wt%.
- The effect of tooth as unit was not statistically significant ($p>0.05$, random effect model).

Discussion

The first null hypothesis stating that block thickness (2 or 6 mm) had no impact on mini-iFT, if the curing time was adapted to provide similar DC of luting RBC, failed to be rejected (p=0.3884). This study confirmed the conclusions of previous works (19, 25, 56) that under specific conditions conventional light-cure restorative composites can be used efficiently as luting composite, and fills the gaps left by those works, as mentioned earlier. All the light transmitted to the luting RBC went indeed 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, i.e. 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 adjustment (extension) of curing time to ensure optimal polymerization conversion 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 a change of state of the resin phase of the RBC (32). The importance of adjusting/extending curing time to optimize DC relies on previous papers (21, 37, 40), showing that even at very low light irradiance, optimal conversion of a conventional restorative RBC using as luting agent can be obtained, provided that a sufficiently long irradiation time is applied. In the present work, 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 sec and 240 sec through a, respectively, 2-mm and 6-mm thick composite block was required. This finding is also in line with more fundamental works 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 polymerization conversion than the opposite, being short curing times at high irradiance (13, 42). The rationale has been explained by a 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 curing light parameters and the loss of light as it travels through the indirect material.

With regards to the objectives of the present work, the LCU must present a large curing tip diameter to cover the restoration and a homogeneous light distribution over the whole restoration surface. Moreover, such a homogenous 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). Solely a slight light-beam inhomogeneity over the tip surface has been reported and attributed to the multiple LEDs chips (3 blue and one violet) present in the LCU, this combined with a coherent light bundle guided towards the curing tip (12). However, this slight light-beam inhomogeneity does not result, at least not reported for materials containing a camphorquinone/amine-based photo-initiator system, in differences in degree of conversion or microhardness over the surface and with depth (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 represents the major factor of 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 was 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 solely valid for the exact combination of parameters set in this study. Specifically with regards to light curing through indirect RBC restorations, as used in this study, the factors influencing the material thickness/light transmittance curve slope are expected to be mostly those related to light scattering by composite-filler particles (hence filler type, size and load, as well as resin-filler refractive index mismatch), and those related to light absorption by pigments (hence material shade) (14). The composite block used in the present work (Katana Avencia, Kuraray Noritake) is filled exclusively with nanofiller (20- and 40-nm particles, 62wt% 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 larger filler particles in high proportion.

The second null hypothesis was rejected, since significant differences in mini-iFT were observed between both luting RBCs tested (<.0001), this in favor of the light-cure composite APX. Previous research comparing adhesive luting of indirect restorations with either light-cure or dual-cure RBCs is limited. The few clinical studies having investigated the matter in posterior restorations have shown minor impact between both luting strategies (27, 28). One twelve-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-cure RBCs as luting agents. However, these works concerned only inlays and onlays of small thickness (<2 mm), while even concerns regarding possible insufficient polymerization of the light-cure materials were raised. To our knowledge, only two studies have investigated this question regarding thicker indirect materials (>4 mm). One measured microhardness of the luting RBC under the indirect restoration, which is not a property enabling direct comparison of bonding quality obtained with light- versus dual-cure polymerization strategies (19). The other, on the contrary, has measured micro-tensile bond strength of 8-mm thick ceramic inlays to dentin (25), and reported lower bond strength using the dual-cure luting RBC as compared to the light-cure 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 enabled light travelling on the side of the inlay, which is not clinically realistic for a large overlay or endocrown, where light can only go through the material bulk. The present work, which happened to use the same RBC APX as in the work by Kameyama et al. (25), nevertheless confirmed the similar trend, showing improved bonding with the high-viscosity restorative RBC. However, it must be mentioned that in this study, the light-cure RBC was preheated at 68°C, initially to increase the flowability of the material for luting purposes.

Pre-heating composite prior to photo-activation was shown to provide greater polymerization conversion, to accelerate polymerization and to reduce curing time (1, 9, 47, 57). Restoration seating and luting RBC-excess removal are clinically easier with (pre-heated) conventional paste-like than flowable RBCs, 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-cure RBC, as unlimited seating/luting time is available. It was also reported that a RBC light-cured through a composite onlay (Signum, Heraeus, Hanau, Germany) of 2-, 3- or 4-mm thickness presented a significantly higher DC (up to 30% for 4-mm thick restorations), when previously pre-heated at 54°C as compared to ambient temperature (1). Our preliminary data obtained by micro-Raman spectrometry conducted in the conditions of this study confirmed a similar trend for APX (data not shown), upon which pre-heating was implemented in the light-cure RBC test group in this study as being advantageous in several aspects.

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 (50wt%). 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 seems to 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 involving usually solely bonding resin or with additional RBC applied on to. Sandblasting removes any contamination by impression material, bacteria, plaque or provisory material, which could potentially hamper bonding of the luting RBC applied on top during the restoration-placement procedure (15). In addition, sandblasting not only micro-roughens the IDS surface, promoting micro-mechanically interlocking, but also exposes the filler particles, making their surface available for surface conditioning and more reactive 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, also the resin phase at the IDS surface still contains chemically active species, more specifically having been disclosed as 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 the exposed filler surfaces.

Trapped free radicals can be detected in dental resin-based materials, among which 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_50wt%, possessing the highest resin content, in which more chemically active groups remained. If the luting composite would primarily bond to the IDS composite via silane treatment and connection to the exposed filler particles, the outcome would show higher mini-iFT for the IDS composite filled at 75wt%. 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 effectively be cleaned by pumice rubbing only, without revealing significant difference in micro-tensile 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 mentioning that it is not instructed in the instructions of PV5 to use 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 were performed to identify a possible impact of applying Tooth Primer (Kuraray Noritake) on the degree of conversion of PV5, in similar conditions than those used for the mini-IFT measurements. 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 work are only valid for a clinical strategy where 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. This method was shown in the past to provide equivalent results to the well-documented micro-tensile tests (43). Potential advantages of interfacial fracture toughness methods over bond strength ones were reported by several groups, the former being described as more reliable, with failures observed at the interface of choice, with lower variations (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 (Figure 1), the use of an interfacial method such as mini-iFT seemed the only appropriate approach, since it enabled to select specifically the interface of interest that was considered important to test. The SEM analyses of the fractured surfaces confirmed the capacity 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

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

Although the findings of the present work can nevertheless not directly be 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 efficient luting when using pure light-cure composite systems. Given the difficulty of mini-iFT measurements and based on the present proof of concept, one option is to perform this optimization of curing parameters by measuring DC of the RBC and to adjust the conditions to reach optimal DC.

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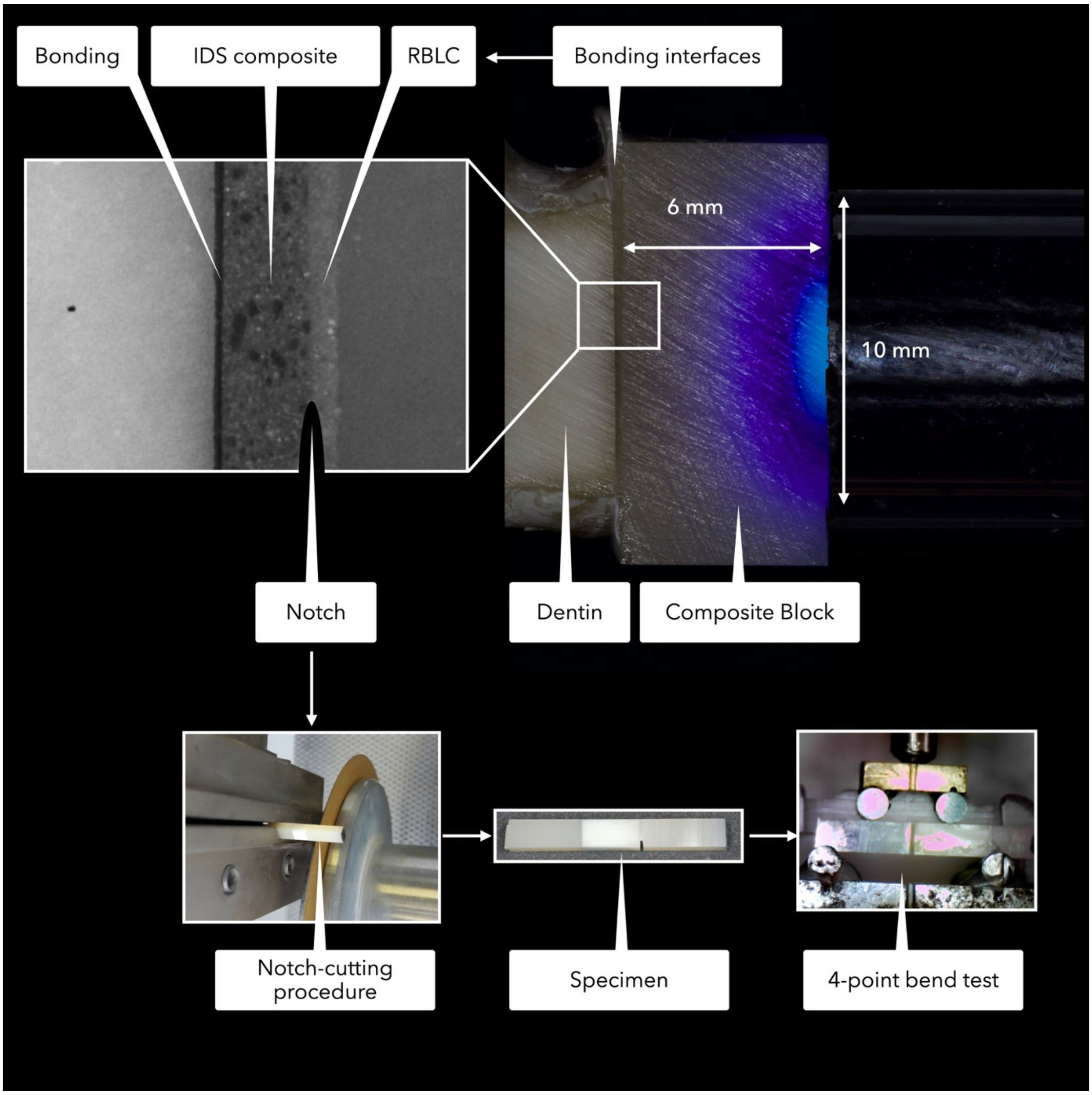

Figure 1 – Illustration of sample preparation procedure. The upper-right picture shows the light tip of the BPG2 light curing unit next to a prepared tooth, and illustrates how light is attenuated as it goes through the composite block. The insert on the upper-left shows the different interfaces under optical microscope, and indicates the interface of interest were a notch was cut. The lower pictures illustrate how the notch was prepared after the teeth were cut into bars, and how the mini-interfacial fracture toughness was tested using an adapted 4-point bend test device. Note that in this example, the luting composite was PV5.

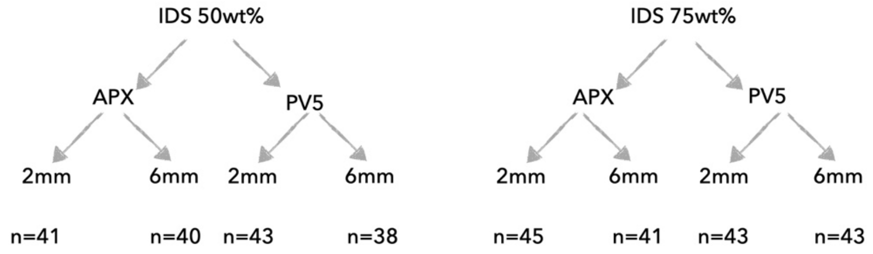

Figure 2 – Summary of the different test groups. Eight third molars were prepared per condition. n = number of testable sticks for each group.

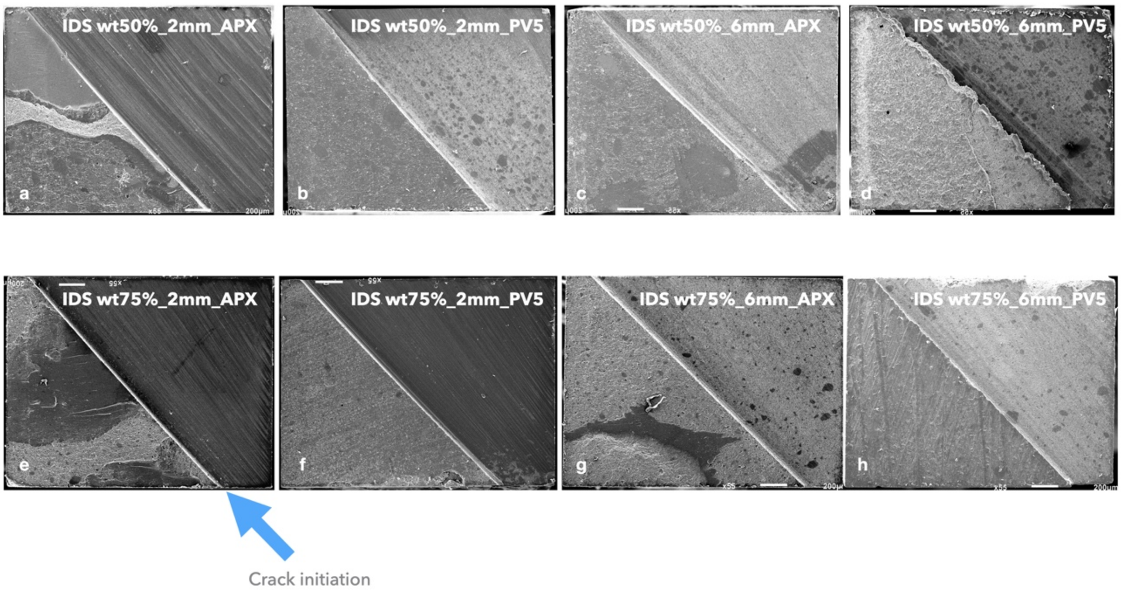

Figure 3 – Representative examples of the fractured interfaces (one for each tested condition – type of IDS_block thickness_RBLC), taken by scanning electron microscopy. **a** and **e** are mixed fractures with <50% interfacial characteristics, so these samples were excluded; **b**, **c**, **f**, **h** are interfacial fractures, hence included; **d** is an interfacial fracture, but between PV5 and the Katana block, so this sample was excluded; **g** is 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 IDS wt75%_2mm_APX, 74% for IDS wt75%_2mm_PV5, 98% for IDS wt75%_6mm_APX and 86% for IDS wt75%_6mm_PV5.

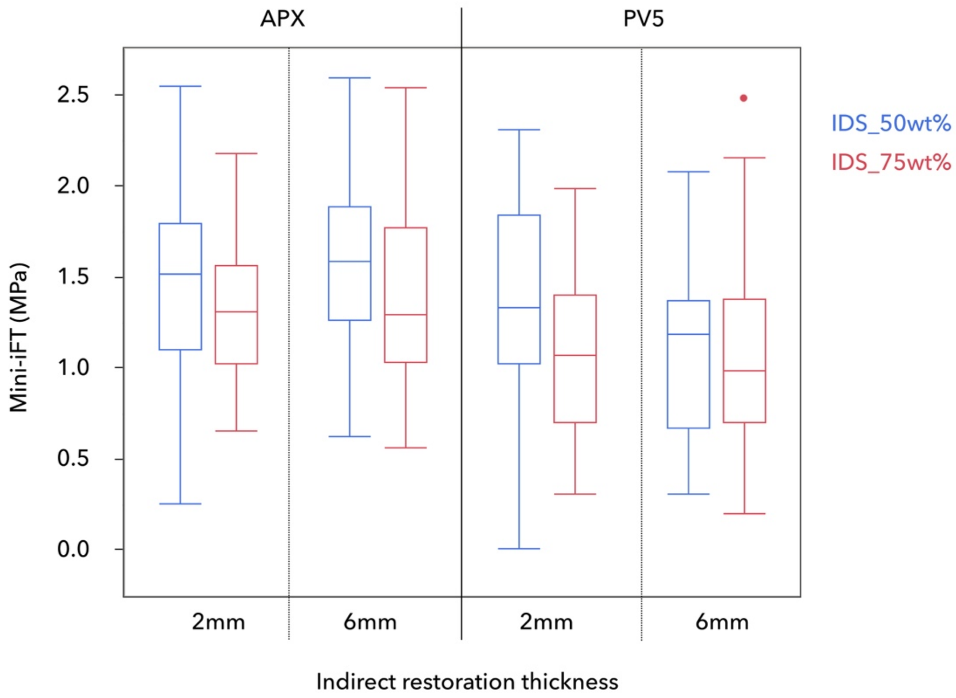

Figure 4 – Mini-IFT data organized in box plots. Values corresponding to APX are on the left, and to PV5 are on the right, each of them either with 2 or 6mm-thick blocks. IDS wt50% values are in blue and IDS wt75% in red.

Table 1 – Mini-IFT results (MPa), standard deviation (SD), number of pre-test failures (ptf), percentage of interfacial fractures (%) and total number testable sticks for each condition (n).

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

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

3-way ANOVA	**Prob. > |t|**
intercept	<.0001*
wt% Fillers IDS	0.0011*
Block thickness	0.3884
RBLC	<.0001*
wt% Filler IDS * block thickness	0.4423
wt% Filler IDS * RBLC	0.8898
block thickness * RBLC	0.0222*