Polymerization Efficacy Affects the Long-term Mini-interfacial Fracture Toughness

Pong Pongprueksa / Jan De Munck / Bruno C. Barreto / Bart Van Meerbeek

Purpose: To evaluate the effect of the type and concentration of photo-initiator on the long-term bonding efficacy to dentin in terms of mini-interfacial fracture toughness (mini-iFT).

Materials and Methods: The bonding efficacy of the one-step self-etch (SE) adhesive Clearfil S3 Bond Plus and four derivative experimental adhesives (Leuven University Bond [LUB]), which were similar in composition except for the (co)photo-initiators (all prepared by Kuraray Noritake), containing 2.0 wt% camphorquinone (CQ) and 2.0 wt% EDMAB (LUB-CQ/amine_high), 0.35 wt% CQ and 0.35 wt% EDMAB (LUB-CQ/amine_low), 2.0 wt% TPO (LUB-TPO_high), and 0.35 wt% TPO (LUB-TPO_low), was measured using a mini-iFT approach after being aged for 6 months and 1 year by water storage at 37°C (control: 1 week mini-iFT). The mini-iFT specimens were loaded until failure in a 4-point bending device to determine the interfacial fracture toughness (K_Ic). All fractured specimens were evaluated using scanning electron microscopy (SEM).

Results: The mini-iFT of Clearfil S3 Bond Plus and the adhesives with high photo-initiator concentrations was not significantly different at 6 months, although it dropped significantly at 1 year. For each storage period, the mini-iFT of the adhesives with high photo-initiator concentrations was not significantly different from that of the commercial adhesive, in contrast to the significantly lower mini-iFT measured for adhesives with low concentrations of photo-initiator. SEM fracture analysis revealed that the adhesives with high concentrations of photo-initiator most frequently failed at the top of the hybrid layer at 1 week, while this failure pattern shifted to the bottom of the hybrid layer after aging.

Conclusion: A one-step self-etch adhesive should contain photo-initiator in a sufficiently high concentration, as this directly improves its immediate and long-term bonding efficacy to dentin. While the mini-iFT remained stable up to 6 month of aging for both the commercial and the adhesive with a high concentration of photo-initiator, the mini-iFT was found to decrease after 1 year of aging.

Keywords: adhesive, bonding, dentin, photo-initiator, fracture toughness, interface.
Simple water storage is commonly used in the laboratory to assess the long-term durability of adhesive-dentin interfaces. Immersion of specimens in water not only directly affects the bond to dentin as well as weakens the bulk properties of the surrounding tissues and substrates, more specifically those of the adhesive, but also of the adjacent dentin and composite. The currently commonly employed microtensile bond strength (μTBS) test measures the strength of the whole test assembly, including the neighboring restorative composite and dentin, rather than solely measuring the strength of the actual interface. Hence, since artificial aging affects all interfacial components, it also increases the test variability and so may interfere with correct interpretation of the results in terms of interfacial bond durability. Recently, we introduced a miniinterfacial fracture toughness (mini-iFT) test as an alternative method to assess bonding efficacy to dentin. Although there is a strong correlation between the μTBS and mini-iFT results, the mini-iFT method not only allows assessment of bonding efficacy in a more standardized way, but it also presents lower variability and higher discriminative power. This mini-iFT method in fact facilitates measuring the strength of the actual adhesive-dentin/enamel interface, for which reason it may be especially suitable for assessing the long-term durability of adhesive-dentin/enamel interfaces.

Therefore, the purpose of this study was to assess the extent to which two polymerization-determining factors, i.e., the type and concentration of photo-initiator, affect the long-term bonding efficacy in terms of mini-iFT of one commercial and four derived experimental adhesives that only differ for the two photo-initiator variables. The 1-week (immediate) mini-iFT was measured first; bond durability was assessed by comparing the mini-iFT measured after 6 months and 1 year of artificial water aging with the immediate mini-iFT.

**MATERIALS AND METHODS**

This study is a follow-up study of a previous project that investigated the effect of the type and concentration of the photo-initiator on the polymerization kinetics and immediate mini-iFT of adhesives. Specimens used for the present study originate from the same teeth as those used in the first study, when only one-third of the specimens were tested at 1 week. Therefore, all materials and techniques presently employed were exactly the same; testing was executed by the same operator. Full methodological details have been described previously, but are repeated briefly below.

Five different adhesives were tested: the commercially available one-step selfetch (SE) adhesive Clearfil S3 Bond Plus (Kuraray Noritake; Tokyo, Japan) and four additional, derived experimental adhesives (Kuraray Noritake), referred to as LUB-CQ/amine_high, LUB-CQ/amine_low, LUB-TPO_high and LUB-TPO_low (LUB: Leuven University Bond). The type and concentration of the photo-initiators are detailed in Table 1.

Fifty noncarious human third molars (collected following informed consent and approved by the Commission for Medical Ethics of KU Leuven, file No. S57622) were stored in 0.5% chloramine T/water at 4°C and used within three months after extraction. The occlusal third of the crown was removed with a diamond saw (Isomet 1000, Buehler; Lake Bluff, IL, USA). The flat dentin surface was wet ground with 320-grit SiC paper (Buehler-Met II, Buehler) to produce a standard smear layer resembling that produced by a conventional diamond bur. Each adhesive was applied following the manufacturer’s instructions (Kuraray Noritake) for the commercial adhesive Clearfil S3 Bond Plus by rubbing the dentin surface for 10 s, followed by 5 s of gentle air drying. The adhesives were then light cured for 10 s using a polywave LED curing light (Bluephase 20i, Ivoclar Vivadent; Schaen, Liechtenstein), employed in high mode with an output of ca 1100 mW/cm²; the radiant exposure was 2.9 J/cm² up to 420 nm and 14 J/cm² above 420 nm, as measured by a MARC Resin Calibrator (BlueLight Analytics; Halifax, NS, Canada). A composite buildup (Filtek Z100, shade A2, lot N459523, 3M ESPE; Seefeld, Germany) was made in layers. The root of the tooth was removed 3 mm below the adhesive-dentin interface, the pulp was removed with a conventional diamond bur, and a similar composite buildup was placed on the root side using the two-step self-etch adhesive Clearfil SE Bond (Kuraray Noritake) and the composite Filtek Z100 (3M ESPE). After 1 week of water storage at 37°C, the specimens were sectioned perpendicular to the interface using a semi-automatic high-speed diamond saw (Accutom-50, Struers; Ballerup, Denmark) to obtain 6 rectangular microspecimens (sticks 1.5 x 2.0 mm wide and 16 to 18 mm long) per tooth.

A single mini-iFT notch tip was prepared under a stereomicroscope (Leica M715; Wetzlar, Germany) precisely at the adhesive-dentin interface using a water-cooled ultrathin 150-μm diamond blade (M1D08, Struers; Ballerup, Denmark; feed speed of 0.015 mm/s, wheel speed 1000 rpm). The notch thickness was < 0.3 mm and encompassed the full adhesive-dentin interface. From each tooth, six microspecimens were obtained and stored in water at 37°C, of which two each were tested at 1 week, 6 months and 1 year. These specimens were transferred to a universal material tester (5848 Micro Tester, Instron; Norwood, MA, USA), putting the specimen upside down (notch tip downwards) in the test fixture. Each specimen was tested in a 4-point bending setup with a crosshead speed of 0.05 mm/min. The outer and inner spans were 10 and 5 mm, respectively. After testing, all fractured surfaces were processed for SEM examination (JSM-6610LV, JEOL; Tokyo, Japan) using the common procedure of fixation, dehydration, and gold-sputter coating to determine the fracture location, crack propagation, and possible specimen imperfections. Finally, the exact dimensions of the mini-iFT notch were measured using a measuring optical microscope (400-NRC, Leitz) at 250X magnification, after which $K_N$ was calculated in MPa·m$^{1/2}$ as also described in detail in the previous study.
The mini-iFT data were statistically analyzed using a linear mixed-effects model. The model was constructed using statistical software (R3.0.1 and nlme package, R Foundation for Statistical Computing; Vienna, Austria). In this model, the two variables “adhesive” and “aging” were included and performed at a critical value of $\alpha = 0.05$. To compare the long-term data to their respective baseline control, specific contrasts, along with a p-value corrected for the number of tests, were calculated (R3.0.1 and multcomp package). All experimental adhesives were compared to the commercial one-step SE adhesive Clearfil S3 Bond Plus which served as control.

### RESULTS

The mini-iFT data are presented graphically in Fig 1 and numerically in Table 2. The mini-iFT of the commercial adhesive Clearfil S3 Bond Plus and the experimental adhesives with high concentrations of photo-initiator remained stable for up to 6 months of water storage, but decreased significantly afterwards, all three to about the same extent. The mini-iFT of the adhesives with low concentrations of photo-initiator was significantly lower at 1 week than the corresponding 1-week mini-iFT of Clearfil S3 Bond Plus and that of the adhesives with high photo-initiator concentrations. The 6-month mini-iFT of LUB-CQ/amine_low significantly de-
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Mini-interfacial fracture toughness (mini-iFT) of the different adhesives at the different time points

<table>
<thead>
<tr>
<th>Adhesive</th>
<th>Aging</th>
<th>Mean mini_iFT(^1)</th>
<th>SD</th>
<th>ptf/n(^2)</th>
<th>Difference from control in MPa m(^{1/2})</th>
<th>p</th>
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<tbody>
<tr>
<td>LUB-CQ/amine_high</td>
<td>1 week</td>
<td>1.79</td>
<td>0.11</td>
<td>0/15</td>
<td>+0.13</td>
<td>0.5</td>
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<td></td>
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<td></td>
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<td>0/17</td>
<td>+0.16</td>
<td>0.338</td>
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<td>LUB-CQ/amine_low</td>
<td>1 week</td>
<td>1.13</td>
<td>0.46</td>
<td>1/16</td>
<td>-0.53</td>
<td>&lt;0.001</td>
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<tr>
<td></td>
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<td>0.67(^*)</td>
<td>0.32</td>
<td>2/13</td>
<td>-1.03</td>
<td>&lt;0.001</td>
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<tr>
<td></td>
<td>1 year</td>
<td>0.66(^*)</td>
<td>0.35</td>
<td>0/15</td>
<td>-0.67</td>
<td>&lt;0.001</td>
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<tr>
<td>LUB-TPO_high</td>
<td>1 week</td>
<td>1.60</td>
<td>0.20</td>
<td>0/16</td>
<td>-0.06</td>
<td>0.952</td>
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<td>6 months</td>
<td>1.73</td>
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<td>0/14</td>
<td>+0.03</td>
<td>0.997</td>
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<td>0/18</td>
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<td>0.45</td>
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<td>8/18</td>
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<td>0.37</td>
<td>7/16</td>
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<td>0.16</td>
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<td>1.23(^*)</td>
<td>0.33</td>
<td>0/15</td>
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</table>

\(^1\)Mini-interfacial fracture toughness \(K_{\text{IC}}\) in MPa m\(^{1/2}\). \(^*\)Group decreased significantly as compared to the 1-week mini-iFT. Any pre-test failures were included as 0 MPa in the statistical analysis. \(^2\)Pre-test failures (ptf) and total number of specimens (n).

increased compared to the 1-week mini-iFT, but then remained relatively stable at 1 year. The lowest mini-iFT was recorded for LUB-TPO_low and was associated with a considerable number of pre-test failures (ptf). Although the mini-iFT was already low at 1 week and 6 months, no further significant decrease in mini-iFT was measured at 1 year.

The schematic diagram in Fig 2 explains the different terms used in the SEM micrographs in Figs 3 to 7, which show representative mini-iFT fractured surfaces of each experimental group. SEM failure analysis revealed that irrespective of the experimental group, the mini-iFT notch tip always failed at the adhesive-dentin interface. Multiple signs of bond degradation were observed at the fractured surfaces. At first, as degradation progressed from the outer specimen border inwards, different failure modes were observed at the outer part of the specimen than at the inner part. This resulted in a triangular area in the center of the specimen, as can be seen most clearly in Figs 3[3a] and 7[3a]. Then, at the outer part of the specimen, the specimen failed consistently along the bottom of the hybrid layer (Fig 3[1a]). At higher magnification, signs of resin degradation were apparent as well. Although for the 1-week mini-iFT specimens, numerous filler particles could be detected (Figs 3[1b] and 7[1c]), they were absent after 6-month and 1-year water storage (Figs 3[2c] and 7[2c]).

For Clearfil S\(^3\) Bond Plus and the adhesives with high photo-initiator concentrations, the crack usually initiated at the interface, but then frequently deviated towards the adhesive layer (Figs 3[1a], 5[1a,2a] and Fig 7[1a,2a]). Within the adhesive layer, brittle fracture patterns were often observed, some radiating crystallite formations closer to the notch tip (Figs 3[1a,2a], 5[1a,2a] and 7[1a]) as well as twist hackle (Figs 3[1a,1c] and 7[1a,2a]), pointing to the crack origin and indicating the propagation direction. These

Fig 2 Schematic diagram explaining the different terms used in the SEM photomicrographs of the fractured mini-iFT specimens.
brittle failure features were more evident in specimens aged for 1 week and 6 months than 1 year. Towards the end of the notch, more often a smooth fractured surface was observed (Figs 3[2a] and 5[2a]), indicating that the failure occurred close to the adhesive-composite interface. At this adhesive-composite interface, small droplets were also observed at high magnification, probably representing water extracted from dentin through osmosis (Figs 3[2c] and 5[2c]).

For the adhesives with low photo-initiator concentrations, the crack almost never deviated towards the adhesive resin, so that the interface could be observed along the whole fractured surface (Figs 4 and 6). Moreover, the hybrid layer and the adhesive resin of LUB-CQ/amine_low appeared very porous (Fig 4[1c,3a,3c]) and even larger droplets were observed within the adhesive resin of LUB-TPO_low (Fig 6[2b,2c,3b,3c]). It is noteworthy that despite the low mini-iFT, the hybrid layer was seldom exposed for LUB-
TPO_low, as a thin resin layer remained to cover the dentin surface of the fractured specimens (Fig 6[1b,1c,3b]).

**DISCUSSION**

This study investigated the bonding efficacy of adhesives that differed in terms of type and concentration of photo-initiator, after long-term water storage using an innovative mini-iFT test protocol. No difference in mini-iFT with aging was observed between the adhesives with high concentrations of photo-initiator and the commercial one-step SE adhesive. The adhesives with low concentrations of photo-initiator resulted in a lower and less reliable mini-iFT, which additionally decreased faster than that measured for the control. Besides differences in mini-iFT, also the failure mode differed ultrastructurally, depending on the type and concentration of photo-initiator.

The mini-iFT remained stable for 6 months for the commercial one-step SE adhesive Clearfil S	extsuperscript{3} Bond Plus and the experimental adhesives with high concentrations of photo-initiator. These results are in line with those of a previous study that demonstrated a stable interfacial fracture toughness of adhesives upon storage in 0.5% chloramine T for 180 days.\textsuperscript{2} Also the CNB fracture toughness of other mild SE adhesives appeared stable after 6-month water stor-
However, in this study, the mini-iFT was found to significantly drop after 1 year of water storage, indicating that the bond degraded rather slowly with time. Apart from a decrease in mini-iFT, water storage also resulted in an increase in test variability; for example, the mini-iFT for the control adhesive Clearfil S 3 Bond Plus and the adhesives with high concentrations of photo-initiator varied between 6.2% and 12.6% at 1 week, and increased to a variance of 11.4% to 13.3% at 6 months and 18.7 to 26.8% after 1 year of aging, respectively. This variance in mini-iFT was clearly higher for the adhesives with low concentrations of photo-initiator at all time points, which may indicate that an insufficient degree of conversion was reached. The fractured surfaces clearly showed morphological signs of interfacial degradation, which appeared to have both endogenic and exogenic causes. Although the literature is not very consistent, endogenic biodegradation of adhesive-dentin interfaces through enzymes is probably more problematic for etch-and-rinse adhesives that produce thick layers of exposed collagen than mild SE adhesives that only partially demineralize dentin and expose collagen fibrils to a much lower degree. Exogenic degradation involving hydrolysis upon water sorption is therefore considered to have primarily degraded the SE adhesive interfaces in this study. Such hydrolytic effects were observable in the form of filler particles that were no lon-

Fig 5  SEM fracture-surface analysis of representative LUB-TPO_high mini-iFT specimens. (1a) SEI view of the fractured dentin-side (D) surface at 1 week; the specimen failed at the actual interface (I) at the notch tip; here a brittle fracture pattern is presented. The crack propagated further along the interface until about the middle of notch, where the crack deviated towards the adhesive resin (Ar) near the notch end. Some resin remnants were found along the notch edge (hand pointer). (1b) BS high-magnification insert (C: composite side) illustrating the brittle fracture area in more detail; small filler particles (bright white spots) were detected within the adhesive layer (Ar). (1c) SEI high-magnification insert (D: dentin side); at this transition zone, the crack shifted direction from within the hybrid layer (Hy) to the adhesive layer (Ar). (2a) SEI view of the fractured dentin-side (D) surface at 6 months; at the outer part of the specimen, the specimen failed at the interface (I), exposing the hybrid layer. Near the notch end, a smooth surface was disclosed, representing the adhesive resin. (2b) BS high-magnification insert (D: dentin side), exhibiting a degradation zone at the outer part of the specimen; the specimen failed at the hybrid layer (Hy), exposing collagen fibrils. Almost no filler particles could be observed inside the adhesive layer (Ar); the numerous black spots represent the holes from which the filler particles were chipped out. (2c) SEI high-magnification insert (C: composite side), showing the smooth surface area near the notch end; many tiny (hand pointer) as well as fewer large droplets (star) were found at the adhesive-composite interface. Filler particles (star) were clearly observable within the composite (Co). (3a) SEI view of the fractured dentin-side (D) surface at 1 year. The degradation progressed from the outer part of the specimen inwards; the specimen failed at the interface (I) near the outer part of the specimen with the adhesive layer (Ar) still being on top at the inner specimen area. (3b) BS high-magnification insert (D: dentin side), exhibiting the outer part of the specimen. Here, unaffected dentin (D) was exposed, as well as the bottom of the hybrid layer (Hy) and the hybrid layer (Hy) at adjacent locations. (3c) SEI high-magnification insert of the notch tip (C: composite side), exhibiting the degradation zone. The specimen failed at the bottom of the hybrid layer (Hy) at the outer part of the specimen, while the adhesive layer (Ar) could still be identified towards the inner part of the specimen.

age. However, in this study, the mini-iFT was found to significantly drop after 1 year of water storage, indicating that the bond degraded rather slowly with time. Apart from a decrease in mini-iFT, water storage also resulted in an increase in test variability; for example, the mini-iFT for the control adhesive Clearfil S 3 Bond Plus and the adhesives with high concentrations of photo-initiator varied between 6.2% and 12.6% at 1 week, and increased to a variance of 11.4% to 13.3% at 6 months and 1 year of aging, respectively. This variance in mini-iFT was clearly higher for the adhesives with low concentrations of photo-initiator at all time points, which may indicate that an insufficient degree of conversion was reached.
ger embedded in resin upon long-term water aging. This could be due to monomers which have been described to be sensitive to hydrolytic degradation. All the adhesives tested contained HEMA, a very hydrophilic monomer that promotes osmosis-driven water sorption, which may have been partially responsible for the water droplets present at the adhesive-composite interface. These droplets were frequently observed in the area near the notch end, where the fractured surface appeared smoother.

Through osmosis, water is sucked from dentin to reach the oxygen-inhibition layer in the adhesive layer; the size and amount of incorporated droplets was found to be higher with a lower degree of conversion. The resin degradation must also have caused the interface to fail near the bottom of the hybrid layer, by which an ultrastructure characteristic of dentin was exposed. This bond degradation clearly progressed from the outside of the specimen inwards. The area exhibiting degradation at the outer frac-
tured surface near the specimen border clearly increased from 6-month to 1-year water storage.

For Clearfil S3 Bond Plus and the adhesives with high concentrations of photo-initiator, the crack frequently deviated towards the adhesive layer (Ar) and even towards the composite (Co) near the notch end. Twist hackle was observed within the adhesive layer around the notch middle (white arrows). Some adhesive resin remnants were observed along the specimen border (hand pointer). (1b) BS high-magnification insert (D: dentin side) exhibiting a resin remnant at the specimen border. The crack propagated from the hybrid layer (Hy) via the top of the hybrid layer (Ht) to the adhesive layer (Ar) and finally to the composite (Co). (1c) BS higher magnification insert (C: composite side), illustrating a brittle fracture pattern in more detail. Small filler particles (bright white spots) were detected within the adhesive layer (Ar). (2a) View of the fractured dentin-side (D) surface at 6 months; the crack initiated at the interface and deviated towards the adhesive layer (Ar) around the notch middle. At the outer part of the specimen, the specimen failed at the interface (I), exposing the hybrid layer. Hackle was observed within the adhesive layer (white arrows). (2b) BS high-magnification insert of the notch tip (C: composite side), exhibiting the degradation zone at the outer part of the specimen. The specimen failed exactly at the bottom of the hybrid layer (Hb), although towards the inner part of the specimen, failure occurred more at the hybrid layer (Hy). (2c) BS high-magnification insert (C: composite side), showing a brittle fracture pattern. Almost no filler particles could be observed inside the adhesive layer (Ar). The numerous black spots represent the voids where the fillers used to be. (3a) View of the fracture dentin-side (D) surface at 1 year. The degradation progressed from the outer part of the specimen towards the inner part of the specimen (white discontinuous triangular line). At the outer part of the specimen, the specimen failed at the interface (I), with scratches from the SiC paper clearly observable. This degradation zone was enlarged as compared to the corresponding interface-failure area observed at 6 months (2a). The inner part of the specimen failed at the interface but remained covered with a micrometer thin layer of adhesive resin (Ar). (3b) BS high-magnification insert (D: dentin side), exhibiting the transition of the outer to the inner part of the specimen; this transition zone involved the hybrid layer (Hy) interspersed with some areas of adhesive resin (Ar). (3c) BS higher magnification insert (C: composite side), exhibiting the transition of the outer to the inner part of the specimen. Collagen fibrils could be observed within the hybrid layer (Hy). No filler particles were found inside the adhesive layer (Ar). The numerous black spots represent the voids where the filler particles debonded.

![Fig 7 SEM fracture-surface analysis of representative Clearfil S3 Bond Plus mini-FT specimens.](image-cropped)
For the adhesives with low concentrations of photo-initiator, the crack always propagated along the interface to the notch end. No compression curls were observed, suggesting a lower strength of the interface complex, as exhibited by the lower mini-iFT measured. A noticeable difference in fracture mode was observed between the low CQ/amine- and TPO-based adhesives. The experimental adhesive that contained CQ/amine in a low concentration exhibited many small porosities within the hybrid layer and adhesive resin. It could be that the photo-initiator concentration was too low to enable the resin to polymerize sufficiently. This may have resulted in monomer elution. An alternative explanation might be that resin was washed out during the dehydration process for SEM; the process consisted of multiple rinsing phases with high concentrations of ethanol. Despite the even lower mini-iFT measured for the TPO-based adhesive, the dentin surface was consistently found to be covered with a very thin layer of resin. A plausible explanation might be that TPO is less hydrophobic than CQ/amine. It can be hypothesized that within a hybrid layer that is richer in water, a water-soluble initiator is more effective, and therefore resin polymerization might have reached a higher DC in the hybrid layer produced by the TPO-based than the CQ/amine adhesive. However within the bulk of the adhesive, TPO polymerization might have been poorer, as a recent study showed that a TPO-based adhesive exhibited no dark-curing reaction at all. In light of this reduced polymerization efficacy, the many hollow spherical structures found throughout the adhesive with a low concentration of TPO are noteworthy. These may have been caused by phase separation; in size and appearance, they are very similar to eggshell-like structures found when glass-ionomer cement is applied on moist dentin.

TPO as an alternative photo-initiator system has been suggested for use in dental adhesives due to its greater efficiency and faster polymerization reaction. In the present study, the use of TPO did not result in any direct benefits with regard to the mechanical properties of the interface complex or its bond durability (Fig 1). The adhesive with a low concentration of TPO performed the worst of all, most likely because of absence of any dark-cure polymerization. Despite their high concentrations of photo-initiator, the experimental adhesives used here are not as effective as the commercial formulation, probably because of the use of highly efficient, proprietary co-initiators. Thus, we concluded that TPO can be used as an alternative photo-initiator system, but without direct benefits in adhesive performance as compared to current adhesives.

CONCLUSION

A one-step SE adhesive should contain photo-initiator in a sufficiently high concentration in order to improve its immediate and long-term mini-iFT. While the mini-iFT remained stable up to 6 months of aging for both the commercial adhesive and those with high photo-initiator concentrations, the mini-iFT was found to decrease after 1 year of aging. The mini-iFT of the adhesives with low photo-initiator concentrations was lower, more variable, and decreased faster upon water-storage aging. Fracture analysis revealed distinct differences in failure mode among the adhesives formulations upon aging, depending on the type and concentration of photo-initiator.

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REFERENCES


Clinical relevance: Photo-initiator type influences the adhesive-dentin bond durability.