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Abstract

This study evaluated the influence of curing devices on marginal adaptation of cavities restored with self-etching adhesive containing CQ and PPD initiators and hybrid composite. Twenty-four class V (3 groups, n=8) with margins located on enamel and dentin were restored with Clearfil SE Bond and Clearfil APX PLT, light-cured with a monowave LED, multiwave LED and halogen light-curing unit (LCU). Marginal adaptation was evaluated with SEM before/after thermo-mechanical loading (TML). On enamel, significantly lower % continuous margins (74.5±12.6) were found in group cured by multiwave LED when compared to monowave LED (87.6±9.5) and halogen LCU (94.4±9.1). The presence of enamel and composite fractures was significantly higher in the group light-cured with multiwave LED, probably due to an increased materials’ friability resulted from an improved degree of cure. The clinician should aware that due to a distinct activation of both initiators, marginal quality may be influenced on the long-term.

Reference

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Marginal integrity of resin composite restorations restored with PPD initiator-containing resin composite cured by QTH, monowave and polywave LED units

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This study evaluated the influence of curing devices on marginal adaptation of cavities restored with self-etching adhesive containing CQ and PPD initiators and hybrid composite. Twenty-four Class V (3 groups, n=8) with margins located on enamel and dentin were restored with Clearfil S3 Bond and Clearfil APX PLT, light-cured with a monowave LED, multiwave LED and halogen light-curing unit (LCU). Marginal adaptation was evaluated with SEM before/after thermo-mechanical loading (TML). On enamel, significantly lower % continuous margins (74.5±12.6) were found in group cured by multiwave LED when compared to monowave LED (87.6±9.5) and halogen LCU (94.4±9.1). The presence of enamel and composite fractures was significantly higher in the group light-cured with multiwave LED, probably due to an increased materials’ friability resulted from an improved degree of cure. The clinician should be aware that due to a distinct activation of both initiators, marginal quality may be influenced on the long-term.

Keywords: Marginal adaptation, Friability, Resin composite

INTRODUCTION

Composites’ shade degradation and an insufficient degree of cure are well known to affect the long-term performance of both resin composites and adhesive systems. To overcome these problems, manufacturers started to highlight the importance of improving the aesthetic appearance of resin composites by limiting the yellowish effect that is due to the presence of camphorquinone (CQ), a canary-yellow powder that is commonly used as initiator. Later, with the increased use of simplified adhesives that usually contain hydrophilic compounds, another problem to face has been the lower degree of polymerization of these simplified adhesives caused by the presence of water. The solution for shade degradation and degree of cure turned up, and it was related to CQ: either it was replaced by alternative initiators or, if irreplaceable, used in conjunction with alternative initiators. These alternatives are called PPD (1-phenyl-1,2-propanedione), MAPO or Lucirin TPO (2,4,6-trimethylbenzoyl-diphenylphosphine oxide) and BAPO or Irgacure (2,4,6-trimethylbenzoyl-phenylphosphine oxide). Some simplified self-etching adhesives contain them, such as Clearfil S3 Bond (Kuraray Noritake Dental, Tokyo, Japan), G-Bond (GC, Tokyo, Japan) or Adhese Universal Bond (IvoclarVivadent, Schaan, Liechtenstein).

It is at this stage where light-curing devices became extremely important, because if CQ has absorption spectra within the range of blue light (470 nm), all these alternative initiators have absorption spectra in the range of ultraviolet and violet light, i.e. around 400 nm. If resin-based materials contain initiators with different absorption spectra, polymerization will be ensured with a light-curing device with an emission range and peak wavelength as similar as possible to the absorption peak wavelength of CQ and all the other alternative initiators. Compared to halogen and monowave LED devices, the advantage of using a multiwave LED is that it offers multiple wavelengths, with a high light output at both wavelengths so that they can consistently cure all dental materials. Therefore, the entire range of proprietary photoinitiators can be activated with such a light source.

1-phenyl-1,2-propanedione, i.e. PPD, has been found to act synergistically with CQ to produce a more efficient photo initiation reaction. This implies that due to improved conversion of double bonds during photo polymerization, dental materials will be optimized in their mechanical properties, biocompatibility and color stability.

Meanwhile, in terms of mechanical behaviour, interesting results have been reported in the literature when materials containing both initiators and light-cured with multiwave LEDs were used. For instance, Cunha Brandt et al. observed more cohesive fractures or surface delamination when their experimental formulation contained a combination of CQ/PPD and was light-cured with a multiwave LED. No explanation was given for this finding, but their SEM images clearly showed a resin composite specimen with a “broken surface” that did not occur with the other materials tested.

While it is well known that a lower rate of polymerization and onset of shrinkage strain will alleviate stresses at the tooth/restoration interface,
it has not yet been determined in which manner an increased rate of conversion, due to the use of a light-curing unit with an emission spectra that perfectly matches those of the CQ/PPD-containing adhesives, might affect the adhesive interface at the marginal adaptation level.

Therefore, the purpose of this study was to evaluate the marginal integrity of restorations made out of a hybrid resin composite and an adhesive system containing CQ and PPD as initiators, light-cured with three light-curing devices with similar radiant emittance: a halogen light-curing unit (LCU), a monowave LED and a multiwave LED. The null hypothesis was that there would be no negative effect of the different curing devices on the marginal adaptation of class V restorations with margins located in enamel and dentin, before and after thermo-mechanical loading.

**MATERIALS AND METHODS**

The restorative materials selected for this study consisted of a 1-component self etching adhesive (Clearfil S® Bond, Kuraray Noritake Dental, Lot #090911, chemical composition: HEMA, Bis-GMA, 10-MDP, silanated silica, di-camphorquinone, PPD, ethyl alcohol, water) and a fine hybrid composite (Clearfil APX PLT, Shade A2, Kuraray, Lot #000345, chemical composition: Bis-GMA, TEGDMA, silanated barium glass filler, silanated silica filler, silanated colloidal silica, di-camphorquinone, catalysts, accelerators, pigments, others). The difference between the experimental groups consisted in the use of the following light-curing units (Table 1):

- a monowave LED (DEMI Plus, Kerr, CA, USA) with a 8 mm diameter light guide (Model: Extended Turbo+) and a radiant emittance from 1,100 to a peak of 1,300 mW/cm² multiple times throughout the curing cycle due to the use of Periodic Level Shifting (PLS) technology,
- a multiwave LED (Valo, Ultradent Products, UT, USA) with an open window and a radiant emittance of 1,000 mW/cm² (curing program: Standard power) and
- a halogen LCU (Swiss Master Light, EMS, Nyon, Switzerland) with a 11 mm diameter light guide and a radiant emittance adjusted to 1,000 mW/cm².

For the three curing units, light output was monitored with both, the radiometer included in the Swiss Master Light device and confirmed with a LED radiometer.

The research protocol for the present study was prepared in accordance to the regulations for the anonymous collection of biological samples approved by the ethical committee of the Canton of Geneva, Switzerland (Human Research Act, article 2, alinea 2) which stipulates that such anonymous tooth collection does not need to be submitted to approval by an ethical committee. Twenty-four extracted anonymous human molars (n=8) were collected for this study. They were stored immediately after extraction and refrigerated in 0.1% thymol solution until the beginning of the tests. Twenty four hours before use they were cleaned with a scaler and pumice, embedded in custom-made specimen holders with their roots in the center using auto-polymerizing resin (Technovit 4071, Heraeus Kulzer, Wehrheim, Germany) and connected to the dentinal fluid simulation device (PAA Laboratories, Linz, Austria). Prior to the holders’ mounting procedure, the root apices were sealed with an adhesive system (Optibond FL, Kerr). To simulate dentinal fluid flow (Fig. 1), a cylindrical hole was drilled into the pulpal chamber approximately in the middle third of the root and a metal tube, with a diameter of 1.4 mm, and was then luted with the same adhesive system. Simulation of dentinal fluid flow would be used, throughout the study, during cavity preparation, adhesive procedures related to cavity filling and thermo-mechanical loading.

Saucer-shaped standardized class V cavities (Fig. 2) were prepared in the dentin for each group. The cavity was filled with the hybrid composite (Clearfil APX PLT, Shade A2, Kuraray, Lot #000345) using a 2-step luting protocol. The groups were as follows:

- Swiss Master Light EMS
- Valo, Ultradent
- DEMI, Kerr

The cured depth of the cavity was measured in mm at 4 locations for each group.

**Table 1** Description of the light curing devices used in this study

<table>
<thead>
<tr>
<th>Curing light</th>
<th>Type</th>
<th>Number and manufacturer</th>
<th>Maximum power density (mW/cm²)</th>
<th>Wavelength (nm)</th>
<th>Tip diameter (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Swiss Master Light EMS</td>
<td>Halogen</td>
<td>M 00042, EMS Nyon, Swiss</td>
<td>3,000, Adjusting digital power density, 390–530</td>
<td>11</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Standard Power 1,000</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>High Power 1,400</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Plasma Emulation 3,200</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Valo, Ultradent</td>
<td>MultiLED</td>
<td>Ultradent Products, South Jordan, UT, USA</td>
<td>1,330</td>
<td>410–500</td>
<td>8</td>
</tr>
<tr>
<td>DEMI, Kerr</td>
<td>MonoLED</td>
<td>Kerr, CA, USA</td>
<td>1,330</td>
<td>410–500</td>
<td>8</td>
</tr>
</tbody>
</table>
2) were prepared by one operator with fine diamond burs (FG 4255/6, Intensiv, Grancia, Switzerland) on the teeth’s buccal cervical area with 50% of the margins located on enamel and 50% on dentin, so that half of the cavity was above the cement-enamel junction and half below. The cavities were cut with uniform dimensions and checked with a periodontal probe (3.5 mm mesio-distal, 3.0 mm occluso-cervical and 1.5 mm deep), then a 1 mm bevel was prepared on enamel margins with a diamond bur (Diatech Dental, Coltène-Whaledent, Altstätten, Switzerland) under water cooling. The margins were finished with 25-micron diamond burs (Diatech Dental) and then, cavity preparations were checked under an optical microscope (Wild M5, Wild, Heerbrugg, Switzerland) at 12× magnification to detect marginal imperfections such as fractures or chipping and cavities were corrected if necessary.

Prior to the application of the adhesive system, no phosphoric acid was used to etch on enamel margins. The self-etching adhesive system was applied according to manufacturer's instructions and light-cured before insertion of the composite layer. Next, resin composite was placed into the cavities in two layers, cervical and occlusal, and light-cured with the different devices for 20 s per layer by keeping the light guide at a near-contact distance (maximum 1 mm) from restorations’ surface. Restorations were then polished with flexible discs of different grit size (SofLex Pop-On, 3M ESPE, Seefeld, Germany).

Thermal and mechanical loading (TML) was applied simultaneously, according to a protocol previously described. The teeth were subjected to 240,000 loading cycles and 600 thermal cycles at 5 and 50 °C. Mechanical stress due to the load cycles was transferred to the center of the occlusal surface with a frequency of 1.7 Hz and a maximal load of 49 N applied by using a natural lingual cusp taken from an extracted human molar. Immediately after completion of the polishing procedure (before loading) and after loading, the teeth were cleaned with rotating brushes and toothpaste. Then, impressions with a polyvinylsiloxane material (President light body, Coltène-Whaledent) were made of each restoration. Subsequently, gold-coated epoxy replicas were prepared for the computer-assisted quantitative margin analysis in a scanning electron
microscope (XL20, Philips, Eindhoven, The Netherlands) at 200× magnification by using a custom-made module programmed with an image processing software (Scion Image, Scion, Frederik, MD, USA). All specimens were subjected to the quantitative margin analysis, examined by a blinded and trained lab technician, and margins were classified according to the criteria detailed in Table 2.

Statistical analysis was performed with specific software (SPSS for Mac, version 21). A 1-way analysis of variance (ANOVA) and Duncan post-hoc test was run to study the effects of the different light-curing units on marginal adaptation of the total margin length, enamel and dentin. The same test was run to detect differences in percentages of non continuous margins due to the presence of enamel/composite fractures. The confidence level was set to 95%.

A post-hoc power analysis was performed to know if sample size was sufficient. To this purpose, statistical power of data at the Total Margin Length (TML) was assessed with a specific software (ClinCalc.com, Post-hoc power calculator). Because in this study percentages of marginal adaptation within the range of 90 to 100% are considered as high-quality margins, power analysis of data above 90% of continuous margins will not be computed. For % of continuous margins at the TML after loading, post-hoc power was of 88.1% when comparing endpoint means of the groups with highest and lowest % of continuous margins (97±3.8 and 84±11.2 respectively), indicating that sample size was appropriate.

Statistical power of data at the Total Margin Length (TML) was assessed with a specific software (ClinCalc.com, Post-hoc power calculator). Because in this study percentages of marginal adaptation within the range of 90 to 100% are considered as high-quality margins, power analysis of data above 90% of continuous margins will not be computed. For % of continuous margins at the TML after loading, post-hoc power was of 88.1% when comparing endpoint means of the groups with highest and lowest % of continuous margins (97±3.8 and 84±11.2 respectively), indicating that sample size was appropriate.

### RESULTS

The results of marginal adaptation, before and after loading, for enamel margins, dentin margins and total margin length are detailed in Tables 3 and 4. Overall, the highest results in terms of continuous margins and the least presence of enamel fractures were reported in the group light-cured by the halogen LCU.

Before loading, no differences were observed between groups \( (p=0.167) \) and at the total margin length the %CM ranged from 98 to 100%, i.e. monowave LED 100%, halogen 99.5±0.9% and multiwave LED 98.9±1.6%. Perfect margins, that is, 100% of continuous margins, were observed in dentin (Table 3). After loading, no significant differences between groups were observed in marginal adaptation on dentin, and results approached 100% (Table 4).

In enamel the situation was different. A significant marginal degradation on enamel was observed in the groups light-cured with the multiwave LED (74.5±12.6) in comparison to the other 2 groups. The predominant failure mode at the enamel margins was the presence of cohesive fractures within enamel as a significant increase of enamel fractures was detected after loading (20.1±10) in respect to the situation before loading (1.4±2.6) as detailed in Table 5.

### Table 3 Percentages of continuous margins for the different groups before thermo mechanical loading on enamel margins, on dentin margins and at the total margin length

<table>
<thead>
<tr>
<th></th>
<th>Enamel before load ( p=0.156 )</th>
<th>Dentin before load</th>
<th>Total margin length before load ( p=0.167 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>MultiLED</td>
<td>98.2 (2.6) A</td>
<td>100 A</td>
<td>98.9 (1.6) A</td>
</tr>
<tr>
<td>MonoLED</td>
<td>100 A</td>
<td>100 A</td>
<td>100 A</td>
</tr>
<tr>
<td>Halogen</td>
<td>99.2 (1.5) A</td>
<td>100 A</td>
<td>99.5 (0.9) A</td>
</tr>
</tbody>
</table>

Differences between groups are represented in capital letters and apply to each column.

### Table 4 Percentages of continuous margins for the different groups after thermo mechanical loading on enamel margins, on dentin margins and at the total margin length

<table>
<thead>
<tr>
<th></th>
<th>Enamel after load ( p=0.004 )</th>
<th>Dentin after load ( p=0.380 )</th>
<th>Total margin length after load ( p=0.007 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>MultiLED</td>
<td>74.5 (12.6) B</td>
<td>95.4 (11) A</td>
<td>84 (11.2) B</td>
</tr>
<tr>
<td>MonoLED</td>
<td>87.6 (9.5) A</td>
<td>99.4 (1.6) A</td>
<td>93.2 (5.5) A</td>
</tr>
<tr>
<td>Halogen</td>
<td>94.4 (9.1) A</td>
<td>99.4 (1.6) A</td>
<td>97 (3.8) A</td>
</tr>
</tbody>
</table>

Differences between groups are represented in capital letters and apply to each column.


Table 5  Percentage of non continuous margins, before and after loading, due to the presence of enamel fractures

<table>
<thead>
<tr>
<th></th>
<th>Enamel fractures before load</th>
<th>Enamel fractures after load</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$p=0.294$</td>
<td>$p=0.000$</td>
</tr>
<tr>
<td>MultiLED</td>
<td>1.4 (2.6) A</td>
<td>20.1 (10) C</td>
</tr>
<tr>
<td>MonoLED</td>
<td>0 A</td>
<td>10.8 (8.5) B</td>
</tr>
<tr>
<td>Halogen</td>
<td>0.8 (1.5) A</td>
<td>0 A</td>
</tr>
</tbody>
</table>

Differences between groups are represented in capital letters and apply to each column.

DISCUSSION

The purpose of this study was to determine if by restoring class V cavities with the same adhesive and resin composite light-cured with three different light-curing sources, i.e. a halogen LCU, monowave LED and multiwave LED, differences would be evidenced at the marginal level. To assess if marginal degradation could be due to early adhesive breakdown or to a poor mechanical resistance, restorations’ marginal adaptation was evaluated before and after thermo-mechanical loading in a chewing simulator combining thermal changes with loading forces. Based on the findings, the null hypothesis stating that there would be no negative effect of the different curing devices on marginal adaptation of mixed class V restorations with margins located in enamel and dentin, before and after thermo-mechanical loading, had to be rejected.

A 1-step self-etching adhesive, Clearfil S3 Bond, was tested in this study because it contains CQ and PPD, i.e. two initiators that absorb light at different wavelengths. Because CQ has an absorption spectrum within the range of 400–500 nm with an absorption peak at 470 nm, we wanted to be sure that this initiator would absorb a maximum of visible light. Therefore, Swiss master light
was selected as the halogen source because the filtered spectral emission matched the absorption profile of CQ very well\textsuperscript{10}. Because PPD co-initiator has an absorption peak at 400 nm\textsuperscript{10} this halogen device would be efficient to activate PPD as well. DEMI, is a monowave LED, with a consistent output of Periodic Level Shifting (PLS) that enables shifting output intensity from 1,100 mW/cm\textsuperscript{2} to a peak of 1,300 mW/cm\textsuperscript{2} multiple times throughout the curing cycle. It operates at a wavelength within the range of 450–470 nm that falls within the maximal absorption of CQ, without initiating PPD. Valo is a multiwave LED that incorporates three different wavelength chips in order to provide sufficient irradiance. As they can operate in both blue and violet regions of the spectrum\textsuperscript{2}, polymerization efficiency increases due to the ability to polymerize other initiators than CQ, if present\textsuperscript{11}. Because PPD co-initiator has an absorption peak at 400 nm\textsuperscript{10} and only those wavelengths where the photosensitizer strongly absorbs are useful for photo polymerization, the different wavelengths are efficient to activate not only CQ, but also PPD. Therefore, any differences in the results would be explained by the influence of the PPD initiator.

In this study, enamel margins were not etched with phosphoric acid prior to the application of the adhesive system. However, the results after loading were around 88 and 94\% of continuous margins for both the monowave LED and halogen groups, respectively (Table 4). The non-differences in marginal adaptation observed between these 2 groups could be explained by a similar behavior of the initiators when light activated by these two units. Halogen units have broad banded regions of the spectrum\textsuperscript{2}, polymerization efficiency increases due to the ability to polymerize other initiators than CQ, if present\textsuperscript{11}. Because PPD co-initiator has an absorption peak at 400 nm\textsuperscript{10} and only those wavelengths where the photosensitizer strongly absorbs are useful for photo polymerization, the different wavelengths are efficient to activate not only CQ, but also PPD. Therefore, any differences in the results would be explained by the influence of the PPD initiator.

Within the limitations of this in vitro study the following conclusions can be drawn:

- Halogen LCU and monowave LED provided with the highest percentages of continuous margin in class V restorations made out of Clearfil S\textsuperscript{3} Bond and Clearfil APX PLT, due to the fact that both units operated at similar wavelengths.
- Light-curing the adhesive system and restorative composite with a multiwave LED adversely affected marginal adaptation in enamel. In view of the high percentages of continuous margin before loading, this was not due to a lack of adhesion, but to a poor mechanical resistance that could not withstand fatigue. A higher degree of cure could have resulted in composites' friability, affecting its brittleness and elastic behaviour at the stiff and brittle enamel margins of class V restorations.
REFERENCES