The effect of light curing units, curing time, and veneering materials on resin cement microhardness

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Abstract Background/purpose: Several factors may affect microhardness of resin cement under veneering materials. The aim of this study was to evaluate the effect of different veneering materials, light-curing units and curing times (20/3, 40/6) on the microhardness of dual-cured resin cement. Materials and methods: We pressed dual-cured resin cement specimens (Clearfil SA cement, 5 mm diameter, 1 mm thick) between two microscopic glass slides covered with transparent polystyrene matrix strips to remove excess material, then irradiated them through a ceramic disc and a composite disc (A2 Esthet X HD, Dentsply, Caulk) with three types of high-power light-curing units as follows: conventional halogen light (quartz tungsten halogen) for 20/40 s, light-emitting diodes for 20/40 s and xenon plasma arc for 3/6 s. The control group specimens were cured under two layer transparent polyester matrix strips (n = 5). After dry storage in the dark (24 h/37°C), we recorded specimens’ Vickers microhardness numbers (50 g load/15 s) and made three indentations on the bottom surface of each one. Data were analyzed using analysis of variance and post-hoc comparisons using Duncan’s test and the Student t test with a significance level of 5%.

Results: Analysis of variance revealed significant differences in microhardness resulting from the different curing units, veneering materials and polymerization times (P < 0.05). The light-emitting diode curing unit produced higher microhardness values compared to the conventional halogen light and plasma arc light sources (P < 0.05). Both veneering materials, ceramic, and composite resin, exhibited significantly lower microhardness values than those of the control group (P < 0.05). Extended polymerization time increased mean surface microhardness values of the resin cement specimens (P < 0.05).

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Introduction

Recent high demand for esthetic restorations has increased the use of composites and ceramics and diminished the use of amalgam. Inlays, onlays, laminated veneers, and ceramo-ceramic crowns are commonly cemented with dual-cured resin cements because light transmission through indirect restorative materials is critical and the chemical reaction theoretically guarantees a satisfactory polymerization. These cements provide adhesion to substrates due to their compatibility with silane agents and adhesive systems, and offer low solubility, easy handling properties, acceptable working time and favorable esthetic results. In addition, resin cements are used to improve the compressive strength of all ceramic crowns compared to glass ionomer and zinc phosphate cement, allowing more effective stress transfer from the restoration to the supporting tooth.

The hardness of a material is the resistance of a solid to local deformation. Hardness is also dependent on the degree of polymerization of the resin matrix. Furthermore, mechanical properties and the biocompatibility of resin cements are directly related to the degree of monomer conversion.

During adhesive cementation procedures, different light sources and veneering materials may affect the polymerization of resin luting agents. Polymerization of these materials can be accomplished with different light sources, including quartz tungsten halogen (QTH), light-emitting diodes (LEDs) and xenon plasma arc (PAC). QTH has the advantage of low cost. Its drawbacks include higher diodes (LEDs) and xenon plasma arc (PAC). QTH has the lifetime of 50,000 hours, while they have an estimated lifetime of about 10,000 hours (in contrast, QTH bulbs have a lifetime of 50–100 hours). PAC curing units emit at higher intensities and were primarily designed to save irradiance time. When using PAC units, the manufacturers recommend 3 s of exposure time to polymerize composite resins with camphorquinone as a photoinitiator system.

Doubts about the effectiveness of light activation of resin cements with different light-curing units (LCUs) and beneath different veneering materials still persist. Peutzfeldt reported that when dual-cure cements are adequately light-activated there is an increase in the degree of conversion compared to dual-cured resin cements subjected to chemical activation alone. This confirms the importance of light exposure to increase the degree of conversion of dual-cure resin cements. One difficulty with indirect adhesive restorations is achieving an adequate degree of polymerization of the resin luting or base material beneath the restoration, especially if using light- or dual-cured resin material. This study evaluated the effect of different veneering materials, different LCUs and two different curing times (20/3 and 40/60) on the microhardness of dual-cured resin cement (Clearfil SA cement, Kuraray, Japan). The null hypothesis was that different values of resin cement hardness would be obtained with different veneering materials, LCUs, and curing times.

Materials and methods

Dual-cure resin cement (Clearfil SA Cement), shade A2, was firmly compressed under a 5-kg load for 3 minutes in a silicon cylindrical mold (1 mm thick and 5 mm in diameter), placed between two glass slides covered by a polystyrene strip to produce a uniform thickness, and then placed against a black background. This background acted as a supporting surface and reduced the reflectivity of the underlying surface toward each specimen. Resin cements were polymerized on veneering materials (5 mm in diameter and 1 mm thick) prefabricated from ceramic material (feldspathic ceramic material) (Duceram Plus; Duceram Dental GmbH & Co. KG, Rosbach, Germany; VITA shade A2) and composite resin (Esthet X HD A2, Dentsply, Milford, USA) used for simulated veneering material.

We prepared the veneering materials 1 week before the experiment with exposure times sufficient to obtain a maximum initial degree of polymerization. Excess resin was removed before polymerization. Two-layer transparent polyester double strips were used as a control group. Three LCUs were used for polymerization of resin cement samples:

- conventional QTH (Hilux curing light, Benlioglu Inc., Ankara, Turkey) LCU for 20/40 s at 450 mW/cm²;
- LEDs (Elipar S10, 3M ESPE, Germany) for 20/40 s at 1200 mW/cm²; and
- PAC (Valo Curing Light, Ultradent Products Inc., USA) for 3/6 s at 4500 mW/cm².

The characteristics of each LCU are shown in Table 1. We measured the power (mW) of the three light sources using a power meter (Hilux Curing Light Meter, Benlioglu Dental Inc., Ankara, Turkey). The light tips were in close contact with either the glass slide or ceramic/composite disc (tip diameter 8 mm) (Fig. 1). After light curing, we stored the specimens dry in light-proof containers in a darkened incubator at 37°C for 24 hours. In order to obtain a smooth planar surface for hardness testing, we polished the bottom surfaces using 400, 600 and 1200 grit SiC papers.

We created 18 groups (n = 5) on the basis of different combinations of veneering materials (double strip, ceramic and composite disc), LCUs (QTH, LED and PAC) and curing times.
polymerization times (20/20/3 s and 40/40/6 s). We obtained microhardness measurements using a microhardness tester (Buehler OmniMet MHT1600-4980T, Buehler, IL, USA), taking three readings with a 50-g load over 10 s on each bottom surface, and converting the average into a Vickers hardness number (VHN) (ISO 6507-1).

Data were analyzed statistically using SPSS 16.0 for Windows (SPSS Inc., Chicago, IL, USA) statistical software. Two-way analysis of variance (ANOVA) was used to compare the variables: LCU, veneering material and curing time. We used one-way ANOVA for intragroup comparisons. Post hoc tests were calculated using Duncan’s test and the Student t test. A confidence interval of 95% was set for all tests ($\alpha = 0.05$).

Results

Mean microhardness values and standard deviations for the top surface dual-cure resins polymerized under veneering materials are given in Table 2.

Two-way ANOVA revealed a statistically significant difference ($P < 0.05$) among the factors investigated (veneering materials, LCUs, and curing time) and also among their interactions ($P < 0.05$). The LED curing unit source produced a higher microhardness value compared to the QTH and PAC light sources ($P < 0.05$). Both veneering materials (ceramic and composite resin), exhibited significantly lower microhardness values than those of the control group ($P < 0.05$). The control groups exhibited significantly higher VHN means than those groups in which the cement was light cured through ceramic and composite restoration.

Microhardness values of resin cement specimens were increased efficiently by extending the polymerization time ($P < 0.05$). The specimens polymerized with LED/40 s beneath double strip produced a significantly higher microhardness than any others. The specimens polymerized with LED/40 s beneath double strip specimens produced a significantly higher microhardness than any other specimens (lower VHN, 77.28 ± 2.98, $P < 0.05$). Those polymerized beneath ceramic specimens with PAC at a 3-s

Figure 1  Schematic illustration of specimen preparation.

Table 1  Curing regimens and conditions.

<table>
<thead>
<tr>
<th>Type</th>
<th>Curing unit</th>
<th>Intensity (mW/cm²)</th>
<th>Time (s)</th>
<th>Total energy (mWs/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Quartz tungsten halogen</td>
<td>Hilux curing light, Benlioglu Inc., Ankara, Turkey&lt;br&gt;&lt;i&gt;Batch number: 3051144&lt;/i&gt;</td>
<td>450</td>
<td>20</td>
<td>9000</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>40</td>
<td>18,000</td>
</tr>
<tr>
<td>Light-emitting diode</td>
<td>Elipar S10, 3M ESPE, Germany&lt;br&gt;&lt;i&gt;Batch number: D-82229&lt;/i&gt;</td>
<td>1200</td>
<td>20</td>
<td>24,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>40</td>
<td>48,000</td>
</tr>
<tr>
<td>Xenon plasma arc</td>
<td>Valo Curing Light, Ultradent Products Inc., USA&lt;br&gt;&lt;i&gt;Batch number: V02640&lt;/i&gt;</td>
<td>4500</td>
<td>3</td>
<td>13,500</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>6</td>
<td>27,000</td>
</tr>
</tbody>
</table>
curing time produced a significantly lower microhardness than any others examined (VHN, 23.22 ± 3.36, P < 0.05; see Table 2). The presence of veneering material resulted in lower VHNs, while a longer curing time resulted in higher VHNs (P < 0.001).

Discussion

This study evaluated the VHN of dual-cure resin cement light-cured through a 1-mm-thick composite or ceramic disc using three different LCUs for two different curing times. The effectiveness of material curing may be assessed directly or indirectly: direct methods, such as infrared spectroscopy, are complex, expensive and time-consuming; indirect methods include visual, scraping, and hardness testing. Many studies have used surface hardness testing because of its relative simplicity and good correlation with the degree of conversion using infrared spectroscopy. Moreover, the hardness test is more sensitive than infrared spectroscopy in detecting small changes in the degree of conversion after the network is cross-linked. Our study used microhardness measurements to estimate the quality of resin curing under veneering materials, since the mechanical properties of resin-based materials can be directly related to the extent of the conversion of the polymer network.

The microhardness of dual-cure resin cements is affected by energy density of the LCUs. Energy density is obtained from the emitted light intensity and curing time. Our results (Table 2) show that lower hardness values were obtained when the resin cement Clearfil SA was light-cured with PAC compared to QTH and LED. Iriyama et al. observed similar results for resin cement Rely X light cured with QTH, LED, and PAC. Light curing with PAC for 3 and 6 s, despite being very fast, does not provide sufficient polymerization of composites, and imperfect polymerization will lead to imperfect properties. Experiments with PAC curing demonstrated that 3 × 3 s light curing at constant high energy densities is sufficient for the polymerization of hybrid resin composites. Lower light energy may affect polymer development, primarily by decreasing the double-bond conversion, since the polymerization process is dependent on radiant exposure delivered to materials. High-intensity lights may favor the formation of more densely cross-linked networks by generating a multitude of polymer growth centers. More densely cross-linked polymers will therefore provide higher hardness outcomes. Soh and Yap have stated that light curing at high intensity would lead to a highly cross-linked polymer chain, and thus to greater hardness. In regions exposed to low energy density, the polymer chain is more linear with higher mobility and lower hardness values.

Adequate polymerization of resin-based luting cement is critical for stability, optimal mechanical features, and the clinical performance of indirect restorations. In addition, maximum bond strength of dual-cured cements is only achieved when light activation is done properly. The degree of conversion in a polymerization reaction is dependent on the energy delivered during light curing, characterized as the product of light intensity and exposure time. Longer light exposure times result in greater composite resin cure depth, conversion degree and hardness. Some have argued that the low irradiation output of LCUs may be compensated for by increasing the irradiation time, without affecting the conversion degree for composites, in such a way that different LCUs can have the same energy density ([mW/cm²] × T). The degree of polymerization with PAC LCU can be compensated for with longer exposure times. PAC techniques require a significant increase in irradiation time when applied to indirect polymerization.

In this study, the LED LCU generally produced higher microhardness values, especially at 40 s polymerization. This probably due to the higher energy density used in these groups. This result is in accordance with a study by Santos et al., who reported higher VHN values with LED LCU compared to QTH LCU. It has previously been reported that LED units are the most efficient ones because they are capable of converting electrical current into the correct

<table>
<thead>
<tr>
<th>Curing lights</th>
<th>Veneering materials</th>
<th>Vickers hardness number (±standard deviation)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>20/3 s</td>
</tr>
<tr>
<td>*Quartz tungsten halogen</td>
<td>Double strips</td>
<td>46.61 (±0.03)⁰,ᴬ</td>
</tr>
<tr>
<td></td>
<td>Composite disc</td>
<td>31.15 (±1.93)⁰,ᴮ</td>
</tr>
<tr>
<td></td>
<td>Ceramic disc</td>
<td>37.86 (±1.99)⁰,ᴬ,ᴮ</td>
</tr>
<tr>
<td>*Light-emitting diode</td>
<td>Double strips</td>
<td>56.68 (±2.21)⁰,ᴬ</td>
</tr>
<tr>
<td></td>
<td>Composite disc</td>
<td>47.44 (±0.31)⁰,ᴬ,ᴮ</td>
</tr>
<tr>
<td></td>
<td>Ceramic disc</td>
<td>40.34 (±1.82)⁰,ᴮ</td>
</tr>
<tr>
<td>*Xenon plasma arc</td>
<td>Double strips</td>
<td>26.64 (±2.17)⁰,ᴬ</td>
</tr>
<tr>
<td></td>
<td>Composite disc</td>
<td>26.93 (±1.69)⁰,ᴬ</td>
</tr>
<tr>
<td></td>
<td>Ceramic disc</td>
<td>23.22 (±3.36)⁰,ᴬ</td>
</tr>
</tbody>
</table>

*For each LCU, means followed by different uppercase letters in the columns differed statistically by Duncan’s test and lowercase letters in the rows differed statistically by the Student t test at the 5% level.
wavelength, similar to the absorption wavelength of camphorquinone. Özyeşil et al, however, observed similar degrees of conversion for resin cement Variolink II light curing with conventional QTH and PAC.\textsuperscript{27}

When light is transmitted through a ceramic or composite, it is absorbed and reflected, losing intensity. Hasegava et al observed a reduction in light when transmitted through laminated veneers during resin cement polymerization. Our study confirmed that the presence of a 1-mm-thick veneering material interposed during curing reduced VHNE.\textsuperscript{28} This finding is also corroborated by other authors.\textsuperscript{14,29} These low hardness figures may be ascribed to light attenuation by the veneering material or the resin cement itself.\textsuperscript{3} They may also be attributed to the different refraction indexes and opacity of the veneering materials, because of their distinct nature (composite and ceramic).\textsuperscript{1}

In our study, there were no significant differences between the veneering materials (composite and ceramic; except for double strips), which involve distinct optical characteristics and compositions. Whereas Esthet X HD is an indirect composite resin, Duceram Plus is a feldspathic porcelain.

The light cure polymerization of resin cements is affected by chemical composition, filler particle size, shade, and the thickness of overlying restorations, as well as light intensity and time of exposure.\textsuperscript{10–32} Favorable polymerization of resin cement is crucial in order to achieve optimal cement properties to prolong the clinical life of the overlying indirect restoration. Additional studies should be conducted to further evaluate light curing using other types of veneering materials, resin cements with different filler loads and monomer and photoinitiator—catalyst compositions.

Within the limits of an in vitro investigation, we concluded that the LED curing unit was associated with the highest hardness values for surface hardness of the dual-cure resin cement (Clearfil SA cement) under veneering materials. LED LCUs may be considered more effective than QTH and PAC LCUs for polymerization of the dual-cure resin cement material (Clearfil SA cement). The presence of simulated veneering restoration material inhibits the polymerization of the underlying dual-cure resin cement material. Increasing the polymerization time had a positive effect on dual-cure resin cement microhardness for all LCUs. In conclusion, prolonged exposure time is necessary in the presence of veneering materials, especially PAC LCUs.

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References


