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Influence of Zirconia Base and Shade Difference on Polymerization Efficiency of Dual-Cure Resin Cement

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Keywords
Zirconia; dual-cure resin cement.

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Previously presented at the European Prosthodontic Association (EPA) 2009 Congress, Innsbruck, Austria.

Funded in part by the Research Fund of Istanbul University.

Accepted September 7, 2010

doi: 10.1111/j.1532-849X.2011.00721.x

Abstract

Purpose: The aim of this study was to investigate the polymerization efficiency of dual-cured resin cement beneath different shades of zirconia-based feldspathic ceramic restorations.
Materials and Methods: Five translucent zirconia (Zirkonzahn) discs (4.0-mm diameter, 1.2-mm height) were prepared. Feldspathic ceramic (1.2 mm) (Noritake Cerabien Zr) in 5 shades (1M2, 2M2, 3M2, 4M2, 5M2) was applied on the zirconia discs. Twelve dual-cure resin cement specimens were prepared for each shade, using Panavia F 2.0 (Kuraray) in Teflon molds (4.0-mm diameter, 6.0-mm height), following the manufacturer's instructions. Light activation was performed through the zirconia-based ceramic discs for 20 seconds, using a quartz tungsten halogen curing device (Hilux 200) with irradiance of 600 mW/cm^2. Immediately following light curing, specimens were stored for 24 hours in dry, light-proof containers. Vickers hardness measurements were conducted using a microhardness tester with a 50-g load applied for 15 seconds. The indentations were made in the cross sectional area at four depths, and the mean values were recorded as Vickers hardness number (VHN). Results were statistically analyzed with one-way ANOVA and Tukey HSD test ($p < 0.05$).
Results: A statistically significant decrease in VHN of the resin cement was noted with increasing depth and darkness of the shade ($p < 0.05$).
Conclusion: Curing efficiency of dual-cure resin cement is mainly influenced by the lightness of the shades selected.

In recent years, the esthetic demands of patients and clinicians have led to the development of high-strength materials such as glass-infiltrated, heat-pressed, and copy-milled ceramics.[1] Bonded all-ceramic restorations provide superior esthetics by allowing diffuse transmission and specular reflectance of light, reproducing a depth of translucency and color mimicking those of natural teeth.[2]

The use of resin cements has been encouraged for all-ceramic restorations because of their low solubility, good esthetics, and high bond strength. To obtain high bond strengths following cementation, selecting the optimal luting agent is required. There are three types of resin cements: self-, light-, and dual-cure, classified according to their polymerization methods.[3]

Self-cure resin cements do not require light to polymerize, and therefore have an advantage when used in deep cavities or under thick restorations; however, mixing of these materials has the risk of entrapping air bubbles that may create voids in the adhesive interface. Also, there may be a slight color shift of the resin cement to yellow when using a tertiary amine catalyst. Light-cure resin cements' ease of use is their major advantage; the working time is not a limitation, and excess luting material can be easily removed prior to polymerization; however, ceramic- or resin-based composite restorations reduce the amount of light reaching the bottom of the cavity and therefore compromise the light activation of the resin cement. Dual-cure resin luting agents were developed in an attempt to combine the desirable properties of self-cure and light-cure resin cements. The chemical polymerizing component was expected to ensure complete polymerization at the bottom of deep cavities, whereas photo-activation allowed immediate finishing after exposure to the curing light.[4]

Polymerization efficiency is the decisive factor when selecting the correct resin cement for different clinical conditions. Adequate polymerization is crucial in obtaining optimal physical properties and a satisfying clinical performance of resin cements. The problem of inadequate polymerization is a low degree of conversion (DC) with a higher residual quantity of double bonds, which causes inferior physical properties and

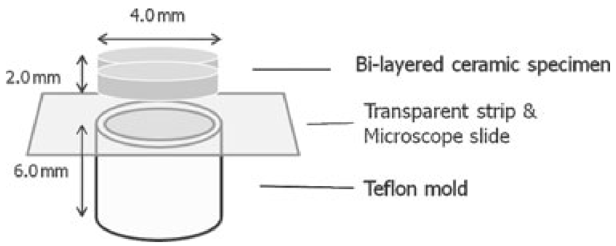

Figure 1 Preparation of resin cement specimens.

raises water absorption and solubility. It has been shown that a high degree of residual monomers may cause pulp irritation and even devitalization.[5]

Several factors may affect the polymerization and mechanical properties of dual-cure resin cements. It has been reported that, when the thickness of restorative materials were increased, the DC and final hardness of most dual-cure resin cements were reduced.[6-9] In addition to the thickness of the restorative material, light transmission properties of the material may result in inadequate polymerization. Light attenuation may affect the polymerization of resin cements used with opaque and even translucent restorative materials. It should also be noted that different brands of dual-cure resin cements have different ratios of light/chemical catalysts; this may result in differences of polymerization efficiency in different commercial brands of dual-cure resin cements.[10-15]

Analyzing the hardness of resin composite has been shown to be a good predictor of DC. Polymerization of the methacrylate monomers in dental resin can form a highly cross-linked matrix, in which a large number of strong covalent linkages between different kinetic chains may transform the molecules into a rigid, very high-molecular-weight material. The cross-linking density could affect the mechanical properties of resin cement. Therefore, investigating the hardness of the material is an accepted method for evaluating physical properties and DC.[16-18]

The aim of this study was to investigate the polymerization efficiency of dual-cured resin cement beneath the different shades of zirconia-based feldsphathic ceramic restorations. The primary hypothesis tested was that shade difference of the restoration affects light polymerizing of the dual-cure resin cement. The secondary hypothesis tested was that dual-cure resin cements require adequate light polymerizing to achieve optimal polymerization.

Materials and methods

Bilayered ceramic disc preparation

Five translucent zirconia discs (4.0-mm diameter, 1.2-mm height) were prepared in the Zirkonzahn CAD/CAM system using zirconia blanks (ICE Zirkonia Translucent 16 mm, Zirkonzahn, Gais, Italy). Feldsphathic ceramic (Noritake Cerabien Zr, Noritake Dental Supply Co. Ltd., Osaka, Japan) (1.2-mm height) was applied onto the fabricated zirconia discs. Five shades of the ceramic were used according to the Vita 3D Master shade guide: 1M2, 2M2, 3M2, 4M2, 5M2.

Each layer of the bilayered ceramic discs was flattened from 1.2 mm down to 1.0 mm by wet grinding on 320-, 400-, 600-, and 800-grit silicon carbide paper and cleaned ultrasonically in distilled water for 5 minutes. The final height of the disc was 2.0 mm (1.0 mm zirconia layer and 1.0-mm feldspathic ceramic layer).

Specimen preparation for Vickers hardness

To evaluate the degree of conversion, Vickers hardness numbers of the resin specimens were evaluated according to ISO 4049. Twelve dual-cure resin cement specimens were prepared for each shade group, using Panavia F 2.0 (Kuraray, Tokyo, Japan). To obtain the specimens, resin cement was placed in Teflon molds (4.0-mm diameter, 6.0-mm height). The mold was placed onto a strip of the transparent film on a glass microscope slide and filled with the test material, according to the manufacturer's instructions. A second strip of the transparent film was put on top, followed by the second microscope slide. The mold and strips of film were pressed between the glass slides to displace excess material. The microscope slide covering the upper strip of film was removed, and the exit window of the light source was gently placed against the strip of film (Fig 1).

Light activation was performed through the zirconia-based ceramic discs for 20 seconds, using a quartz tungsten halogen curing device (Hilux 200, Benlioglu, Turkey) with irradiance of 600 mW/cm^2. The curing light was monitored with the built-in light meter after polymerizing each resin specimen. Following light polymerization, specimens were stored in dry, light-proof containers for 24 hours. To obtain a smooth surface for hardness testing, the specimens were embedded in cold-curing methylmethacrylate using cylindrical molds (10-mm diameter, 6-mm height) and transversely wet-flattened with 320-, 400-, 600-, and 1200-grit SiC papers.

Vicker's hardness measurements were conducted using a microhardness tester (Leica VMHT, Leica, Solms, Germany) with a 50-g load applied for 15 seconds. The indentations were made in the cross-sectional area at four depths (100, 300, 500, 700 μm). The top surface of each resin specimen was marked with one indentation, to be used as a reference point for measurement. Using the gauges on the handles for x- and y-axes, Vickers hardness number (VHN) values at 100, 300, 500, and 700 μm from the first indentation on the top were recorded (Fig 2). The measurements were also cross checked on the PC monitor connected to the tester device. The mean values were recorded as VHN.

Statistical analysis

The VHN values for different shades of ceramics and for different depths of measurement were analyzed with two-way ANOVA with significance level at $p < 0.05$. The differences between VHN values were analyzed by LSD test, with a $p < 0.05$ significance level.

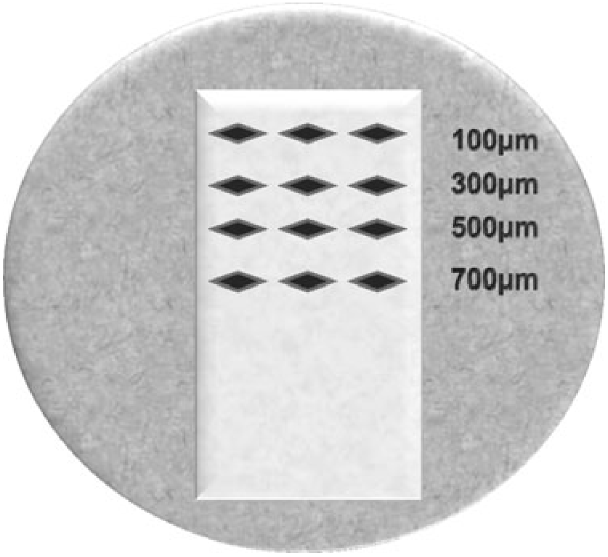

Figure 2 Vickers microhardness test measurement depths.

Results

The comparison and the significance of VHN value differences between shade groups are shown in Table 1 and Fig 3. In each shade group, VHN mean values decreased as the measurements were taken further from the irradiation source.

When the group with the greatest lightness (1M2 shade) was evaluated, the Vickers hardness mean values were not significantly different down to 500-μm depth. The difference of VHN mean values between 100 and 500 μm and between 100- and 700-μm subgroups were statistically significant. Between 500 and 700 μm, farthest from the light source, there was no significant difference.

For the 2M2 shade group, there was no significant difference in the 100- and 300-μm subgroups; however, as the distance from the light source increased, the decrease of VHN mean values in the 300-, 500-, and 700-μm subgroups were statistically significant. In the remaining groups (3M2, 4M2, 5M2 shades), the differences of VHN mean values for the 100-, 300-, 500-, and 700-μm subgroups were statistically significant. As the VHN values were measured further from the light source, the hardness of the resin cement decreased significantly for each subgroup.

Table 1 Vickers hardness mean values. Means followed by different capital letters in the same line, and small letters in the same column, were statistically different at $p < 0.05$

	100 μm	300 μm	500 μm	700 μm
1M2	65 ± 7 ^{A,a}	59 ± 2 ^{A,a}	55 ± 6 ^{B,a}	53 ± 9 ^{B,a}
2M2	59 ± 5 ^{A,b}	52 ± 5 ^{A,b}	45 ± 7 ^{B,b}	34 ± 8 ^{C,b}
3M2	55 ± 5 ^{A,b}	43 ± 5 ^{B,c}	35 ± 6 ^{C,c}	29 ± 5 ^{D,b}
4M2	44 ± 3 ^{A,c}	34 ± 4 ^{B,d}	20 ± 2 ^{C,d}	17 ± 4 ^{C,c}
5M2	41 ± 5 ^{A,c}	28 ± 5 ^{B,e}	25 ± 4 ^{C,d}	18 ± 4 ^{D,c}

Discussion

Different factors, including the intensity of light transferred through the restoration, determine the mechanical properties of dual-cure resin cements. The results support acceptance of the research hypotheses that shade difference of the restoration affects light polymerization of the dual-cure resin cement, and dual-cure resin cements require adequate light polymerization to achieve optimal polymerization. Several studies reported that most dual-polymerized luting agents are extremely dependent on photoactivation, and chemical polymerization alone does not ensure complete polymerization of the luting agent. Most all-ceramic systems, especially zirconia restorations, hinder light polymerization of dual-cure resin cements.

In this study, in order to evaluate the degree of polymerization, the hardness of the material was used as a parameter. The direct evaluation of the degree of polymerization of photoactivated, resin-based composites by spectroscopic techniques is not easily accomplished. Hardness tests provide a good correlation with the degree of polymerization; therefore, the indirect evaluation using hardness is widely accepted.[19]

In this study hardness changed as a function of cement depth. Although film thickness of 300 to 700 μm might not be suitable for luting purposes, the in-depth polymerization of luting agents may indicate their polymerization potential. In a clinical situation, the light attenuation effect promoted by any indirect restorative could be affected negatively by different irradiance levels of polymerization devices, as well by different shades, opacities, and thicknesses of the intervening restorative material. Materials with higher potential for in-depth polymerization would be preferable for situations when low light energy is available.[19]

This study aimed to investigate the importance of light polymerization in dual-cure resin cement. To evaluate this, the behavior of the dual-cure resin cement under incident light should be observed. The initial light exposure causes a rapid increase in conversion of the resin, resulting in a very viscous gel. This rapid increase in viscosity hinders the migration of active radical components that would be responsible for further chemically induced polymerization. Therefore, the duration of inhibition and the level of initial conversion caused by the light exposure are highly influential factors upon the final polymerization of a dual-cure resin.[19,20]

Zirconia is an opaque material, and even translucent zirconia ceramics have significant light attenuation effect. In a study by Hofmann et al,[4] where resin cement was irradiated through 2.5-mm leucite-reinforced glass ceramic, dual polymerization produced better mechanical properties than photoactivation alone. Dual polymerization compensated for irradiation through porcelain, at least for most parameters/materials; however, these results contradict the results of our study, where the light attenuation effect of the ceramic influenced the polymerization of the dual-cured resin cement negatively. The reason for this contradiction may be the difference between the light-attenuation effect of leucite-reinforced glass ceramic compared to the feldspathic ceramic/translucent zirconia bilayered specimen in this study.[4]

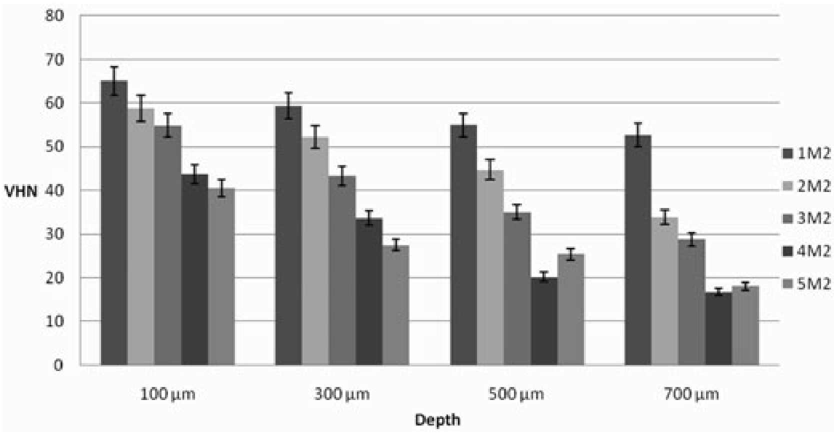

Figure 3 Vickers hardness means and standard deviations for depths of measurement ($p < 0.05$).

Rueggeberg[21] reported that incident light is attenuated with increasing distance from the irradiated surface, as a result of absorption and scattering effects promoted by fillers and resin components. Rueggeberg also reported that about only 25% of the light energy hitting the top surface of the composite is available at 1-mm depth. These evaluations were in accordance with the results of our study, where the VHN values decreased as the distance from the light source increased. This can be interpreted as a decrease in polymerization taking place as the incident light was attenuated.

The additional self-polymerization of the dual luting agent was generally inefficient to compensate for the reduced quantity of light energy reaching the bottom layer in achieving a similar hardness to the top layer. This finding was in accordance with the findings of El-Mowafy et al,[22] who reported that dual-cured cements were dependent on light exposure to achieve optimal properties. In addition, Aravamudhan et al showed that depth of polymerization reduced linearly with increasing distance from the resin composite surface.[19,23]

The data from this study indicate that dual-cure resin cements are extremely dependent on photoactivation, and chemical polymerization alone does not ensure complete polymerization of the resin cement. Zirconia ceramics provide superior mechanical properties; however, their opacity is high. Therefore, light transmission is low compared to other all-ceramic systems. Inadequate polymerization of dual-cure resin cements may occur, especially with darker shades of zirconia-feldspathic ceramic restorations. Clinicians should be aware of the effects of subtle color changes on the polymerization efficiency of the dual-cured resin cements. This study supported the findings of similar studies[19-23] stating dual-cure resin cement requires adequate light polymerization, and use of these materials under 2 mm or thicker restorations requires using either dual-cure cements with longer light curing than recommended by the manufacturers, or using self-cure resin cements or a dual-cure resin cement with a higher chemical curing component. Further studies are recommended for evaluating longer polymerization times and different brands of resin cements.

Conclusions

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

1. Higher color value of the ceramic affected the light transmission negatively.
2. Less light transmission resulted in a lower degree of conversion in the material. In clinical applications, under ceramic restorations with darker shades, dual-cured resin cements may result in inadequate polymerization. It should also be noted that, in some patients, in order to achieve the required natural esthetic result, much darker shades than the ones used in this study may be needed.
3. Further studies are recommended to evaluate polymerization efficiency of other dual-cured resin cements, using different light sources, with prolonged polymerization times beneath different ceramic materials and shades.

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