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Early Hardness and Shear Bond Strength of Dual-cure Resin Cement Light Cured Through Resin Overlays With Different Dentin-layer Thicknesses

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Clinical Relevance

Reducing the dentin-layer thickness while increasing the translucent-layer thickness of resin inlays increases the photopolymerization of dual-cure resin cement, thereby increasing the early bond strength of resin inlays to dentin.

SUMMARY

The purpose of this study was to investigate whether dentin-layer thickness of resin overlays could affect the early hardness and shear bond strength of dual-cure resin cement (DCRC, RelyX ARC) after light curing with light curing units (LCUs) of various power densities: Optilux 360 (360), Elipar Freelight 2 (FL2), and Elipar S10 (S10). Resin overlays were fabricated using an indirect composite resin (Sinfony) with a dentin layer, an enamel layer, and a translucent layer of 0.5 mm thickness each (0.5-0.5-0.5) or of 0.2 mm, 0.5 mm, and 0.8 mm thickness (0.2-0.5-0.8), respectively. The DCRC was light cured for 40 seconds through the overlays, and surface hardness and shear bond strength to bovine dentin were tested 10 minutes after the start of light curing. Surface hardness was higher when the DCRC was light cured through the 0.2-0.5-0.8 combination than when the DCRC was light cured through the 0.5-0.5-0.5 combination with all LCUs. The ratio of upper surface hardness of DCRC light cured through resin overlays relative to the upper surface hardness of DCRC light cured directly was more than 90% only when the DCRC was light cured with S10 through the 0.2-0.5-0.8 combination. The shear bond strength value was higher when the DCRC was light cured with S10 through the 0.2-0.5-0.8 combination than when light cured with S10 through the 0.5-0.5-0.5 combination. This study indicates that reducing the dentin-layer thickness while increasing the translucent-layer thickness of

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resin inlays can increase the photopolymerization of DCRC, thereby increasing the early bond strength of resin inlays to dentin.

INTRODUCTION

Resin cements are the materials of choice for the adhesive luting of resin inlays. Resin cements provide a strong and durable resin bond with high retention,[1] marginal adaptation, and microleakage prevention.[2] Resin cements also increase the fracture resistance of the restored tooth and the restoration itself.[3] The compositions and characteristics of resin cements are similar to those of conventional composite resins,[4] and they are available in chemical-cured, light-cured, and dual-cured formulations. Many of the available resin cements are dual-cured resin cements (DCRCs), and they polymerize when the base and catalyst components are mixed and when they are subjected to the curing light from a light source.[5] DCRCs offer the advantages of extended working time and controlled polymerization,[6] and the chemical activators ensure a high degree of polymerization. However, most DCRCs still require light curing and demonstrate inferior hardness when light curing is omitted.[5,7-9]

During adhesive luting of resin inlays, only the external walls of the cement interface can benefit from direct light curing because they are readily accessible to the dental curing light. Polymerization of the DCRC at the internal walls, for example, the pulpal floor or the axial wall, relies more extensively on the chemical component of the curing mechanism[5] because the curing light is attenuated by the tooth structure or the resin inlay itself.[7] Chan and Boyer[10] and Barghi and McAlister[11] investigated the hardening of light-cured resin cements through porcelain and found that the thickness and shade of porcelain could affect the hardness of the cement. Blackman and others[12] found that the polymerization of resin cements beneath ceramic inlays was proper up to 3 mm distance from the tip of a standard curing light. Hasegawa and others[7] studied the hardening of three DCRCs under resin inlays and reported that chemical curing alone did not completely harden the DCRCs when the curing light was attenuated by the tooth structure and the restoration material. Park and others[13] reported that as much as 120 seconds of curing time using a high-power density halogen light-curing unit (LCU) was needed for proper curing of the DCRCs under the 1.5 mm Targis overlay.

Light intensity decreases as a function of depth, because the translucency decreases as the thickness of the composite resin increases, and the refractive index of the composite resin limits the speed of light transmission.[14] Thorough light curing of DCRCs, therefore, depends on light penetration to a desired depth through resin inlays that may prevent such a penetration.[15] LCUs must emit radiations between 410 and 500 nm, and the power density must be at least 300 mW/cm^2 for proper photopolymerization of the composite resin.[16] To achieve fracture resistance of resin inlays, the minimum thickness of resin inlay should be 1.5 mm,[13] and at least 300 mW/cm^2 of curing light is needed for proper light curing of the DCRCs under the resin inlays. Furthermore, resin inlays are fabricated with more than one layer; the dentin layer, the enamel layer, and the translucent layer are used simultaneously. Unfortunately, there are no studies evaluating how the thickness of each layer affects indirect composite resins, although the thickness of each layer affects the curing light penetration through resin inlays[17,18] and thereby the photopolymerization of DCRCs under resin inlays.

Some studies have evaluated the hardness of DCRCs light cured through various indirect restorations, such as ceramics, zirconia, and composite resins of a single shade.[12,15,19-23] However, there are no studies on the shade combinations of a resin inlay simulating the clinical situation including all the dentin, enamel, and translucent layers. In our previous study with the same indirect composite resin used in this study, we measured the power density of LCUs through resin overlays with various layer thickness combinations. The thickness of the dentin layer was decreased from 0.5 mm to 0.1 mm, the thickness of the enamel layer was kept unchanged at 0.5 mm, and the thickness of the translucent layer was increased from 0.5 mm to 0.9 mm and vice versa to maintain the resin overlay thickness of 1.5 mm. Higher-power density was measured through resin overlays with dentin-layer thickness of less than 0.2 mm and increased translucent-layer thickness in all LCUs.[17]

Therefore the purpose of this study was to investigate whether 1.5-mm-thick resin overlays with dentin-, enamel-, and translucent-layer thicknesses of 0.5 mm each, or 0.2, 0.5, and 0.8 mm could affect the photopolymerization of the DCRC. At first, the DCRC was light cured through two layer thickness combinations of the resin overlays and microhardness was measured. Then, to simulate the clinical situation of resin inlay cementation, the DCRC was light cured through the resin overlays

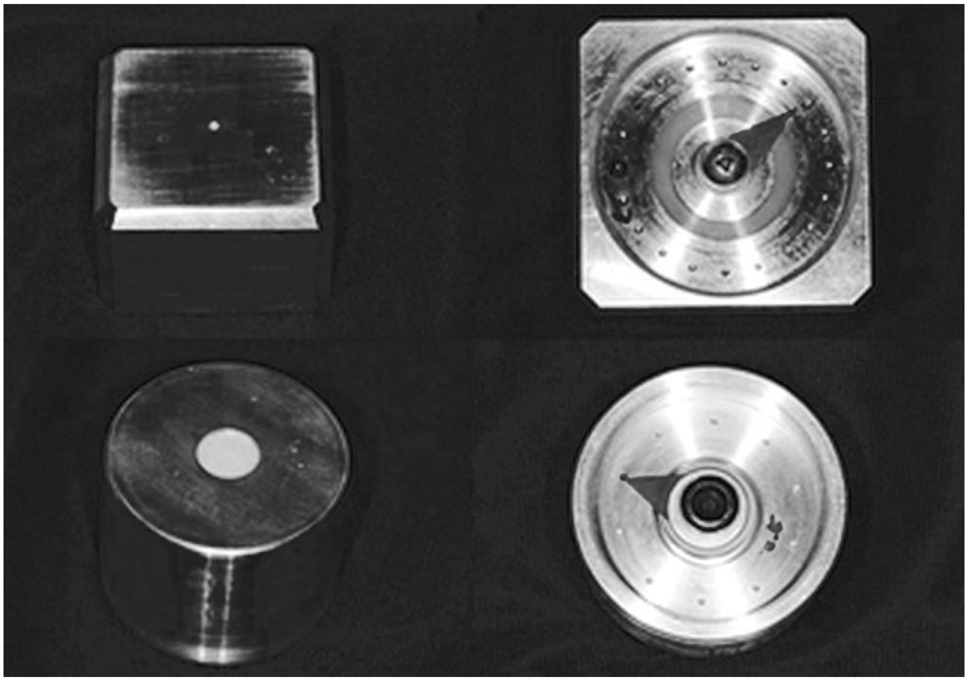

Figure 1. *Photograph of custom-made metallic molds with a movable Teflon plate and a bolt attached to the opposite side of the Teflon plate.*

and the shear bond strength (SBS) to bovine dentin was investigated.

METHODS AND MATERIALS

Resin Overlay Fabrication

We fabricated 1.5-mm-thick resin overlays in 15-mm diameter for the microhardness test and 3-mm diameter for the SBS test. Two layer combinations of resin overlays were fabricated according to our previous study:[17] resin overlays with dentin-layer thickness of 0.5 mm, enamel-layer thickness of 0.5 mm, and translucent-layer thickness of 0.5 mm (0.5-0.5-0.5 combination); and resin overlays with dentin-layer thickness of 0.2 mm, enamel-layer thickness of 0.5 mm, and translucent-layer thickness of 0.8 mm (0.2-0.5-0.8 combination).

Two custom-made cylindrical metallic molds with a hole (15 mm or 3 mm in diameter and 30 mm in depth) at the center were used to fabricate the resin overlays (Figure 1). A flat Teflon plate was inserted into the hole, and a bolt was attached to the opposite side of the Teflon plate, so that the empty space in the hole could be adjusted by rotating the bolt. By rotating the bolt 360° counterclockwise, the Teflon plate could be moved in a downward direction thereby rendering a 1-mm deep empty space in the metallic mold. The rotation of the bolt was marked in 10 steps such that one step corresponded to a downward movement of the Teflon plate by 0.1 mm (Figure 1).

Three layers of Sinfony indirect lab composite (3M ESPE, Seefeld, Germany) were used to fabricate the resin overlays: A2 dentin layer, E2 enamel layer, and T1 translucent layer. To fabricate resin overlays with each layer of 0.5 mm thickness, the Teflon plate in the mold was lowered by 0.5 mm, and the empty space was filled with the A2 dentin layer. Then, the upper surface of the mold was covered with a transparent polyester film and a glass slab to press the surface to remove the excess composite resin. The dentin layer was light cured with a light-emitting diode (LED) LCU (Elipar FreeLight 2 [FL2], 3M ESPE, St Paul, MN, USA) for 5 seconds. After removing the glass slab and the polyester film, the dentin layer was light cured for 20 seconds using an overlapping curing procedure to ensure that every part of the dentin layer was light cured. The Teflon plate with light-cured dentin layer was lowered by another 0.5 mm and the E2 enamel layer was filled into the empty space and light cured as described earlier. The same procedure was repeated with the T1 translucent layer to fabricate the resin overlay of 1.5-mm thickness.

The resin overlays with dentin-layer thickness of 0.2 mm, enamel-layer thickness of 0.5 mm, and translucent-layer thickness of 0.8 mm were fabricated as described previously by controlling the empty

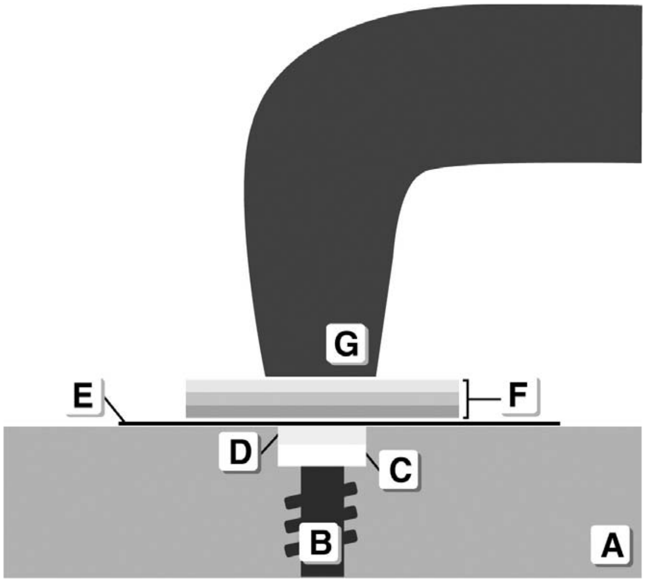

Figure 2. *Schematic diagram of the DCRC specimen fabrication for the microhardness measurement. (A): Metallic mold. (B): Bolt. (C): Teflon plate of 3-mm diameter. (D): DCRC. (E): Polyester film. (F): Layered resin overlay of 15-mm diameter. (G): Light guide of the LCU.*

space made by the Teflon base. Seven resin overlay specimens of each layer thickness combination were fabricated in 15-mm diameter and in 3-mm diameter (n=7).

Microhardness of DCRC Light Cured Through Resin Overlays

To light cure the DCRC in one curing procedure, the custom-made metallic mold with a 3-mm diameter hole was selected as the smallest tip diameter of the LCU was 7.5 mm. The Teflon plate was lowered by 1 mm, and the DCRC (RelyX ARC; 3M ESPE, Seefeld, Germany) was mixed according to manufacturer's instructions and inserted into the empty space. The upper surface of the mold was covered with a polyester film to separate the DCRC and the resin overlay. Previously fabricated resin overlays of 15 mm in diameter were used to cover the DCRC and pressed with a glass slab to control the resin cement thickness to 1 mm. After removing the glass slab, the DCRC was light activated through the resin overlays with LCUs for 40 seconds according to the manufacturer's instructions (Figure 2). Three different LCUs were used to light cure the DCRC: a halogen LCU (Optilux 360 [360]; Demetron, Danbury, CT, USA) with a power density of 530 mW/cm^2, and two LED LCUs (FL2 and Elipar S10 [S10], 3M ESPE) with a power density of 1040 mW/cm^2 and 1340 mW/cm^2, respectively. The power density of the LCUs was measured with a handheld dental radiometer (Cure

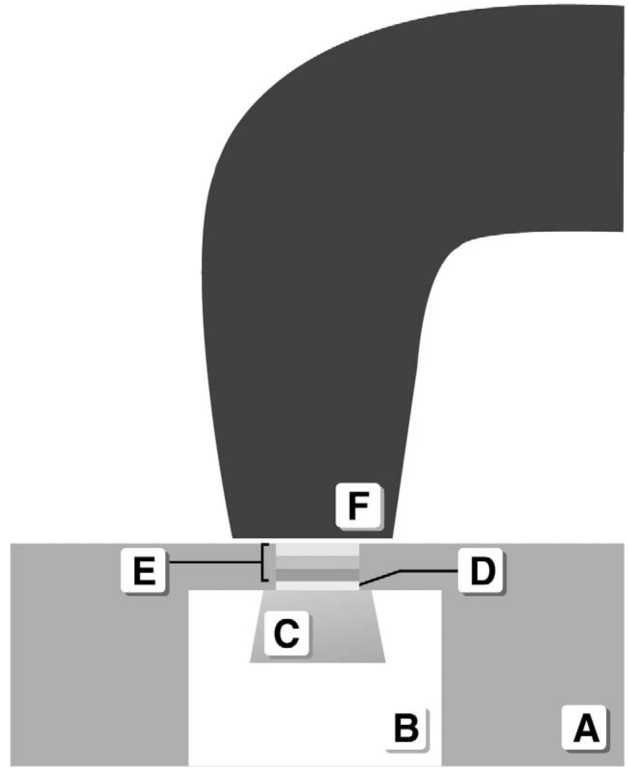

Figure 3. *Schematic diagram of specimen fabrication for the SBS test of DCRC to bovine dentin. (A): Metallic mold. (B): Acrylic resin. (C): Bovine dentin. (D): DCRC. (E): Layered resin overlay of 3-mm diameter. (F): Light guide of the LCU.*

Rite, Kerr, Milford, OH, USA). Ten minutes after the start of light curing, the DCRC was removed from the mold and Vickers microhardness was tested with a microhardness tester (MHT-10, Anton Paar, Graz, Austria) with 100 g load for a 10-second dwell time at three points forming a small triangle in the center of the upper surface of the resin cement; the mean Vickers hardness number (VHN) was recorded. As a control, the DCRC was light cured directly without resin overlays for 40 seconds and the microhardness was measured as described previously. Therefore, nine experimental groups (two resin overlays with different layer combinations and the control group and three LCUs) were tested with seven DCRC specimens each (n=7). The ratio of the upper surface hardness of the DCRC light cured through composite resin overlays relative to the upper surface hardness of the DCRC light cured directly (control) was calculated for the hardness ratio.

SBS of DCRC to Bovine Dentin Light Cured Through Resin Overlays

Freshly extracted bovine incisors were stored in distilled water and the water was changed every two

Table 1: *Summary of Two-way ANOVA for Main Factors (Layer and LCU) and Their Interactions for the VHN*

Source of Variation	Sum of Squares	*df*	Mean Square	F	Significance
Layer combination of resin overlay	1529.391	2	764.695	1012.803	<.001
LCU	772.831	2	386.416	511.789	<.001
Layer combination of resin overlay * LCU	168.689	4	42.172	55.855	<.001
Error	40.772	54	0.755		
Total	108,088.574	63			

days according to ISO/TS 11405:2003(E).[24] The roots were removed with a low-speed disc, leaving only the crown portion. Each crown portion was cut parallel to the labial surface to expose the dentin and then ground flat with wet 600-grit silicon carbide paper. The exposed dentin was sectioned with a low-speed microsaw (Topmet, Norderstedt, Germany) under water spray to a size of 5 × 5 mm. Placing the exposed dentin surface facing the bottom of the mold, bovine dentin was inserted into a metallic mold with a 22-mm inner diameter and a 15-mm depth and chemical-cure acrylic resin (Ortho-jet acrylic, Lang Dental Manufacturing, Wheeling, IL, USA) was poured into the mold. The embedded dentin specimen was removed after curing, and the exposed dentin was wet bonded using 37% phosphoric acid and SingleBond (3M ESPE) application followed by light curing for 20 seconds.

The embedded dentin specimen was inserted into another metallic mold with a 22-mm inner diameter and a 15-mm depth. In the center of the mold, a perforating hole with a 3-mm diameter and 1.7-mm thickness was prepared from the inner side to the outer surface of the mold. With the dentin surface exposed through the 3-mm diameter hole, the DCRC was applied to the dentin surface in 0.2-mm thickness into the hole according to the manufacturer's instructions. Previously fabricated resin overlays of 1.5-mm thickness and 3-mm diameter were inserted into the hole with the dentin layer facing the DCRC and pressed with a glass slab to control the cement layer to 0.2-mm thickness. The glass slab was removed and the DCRC was light cured for 40 seconds according to the manufacturer's instructions (Figure 3). Ten minutes after the start of light curing, the specimens underwent SBS testing with a universal testing machine (Z020, Zwickl, Ulm, Germany) with 500 N load cell at 1 mm/min crosshead speed until fracture; the acquired bond strengths were converted to megapascals (MPa). Therefore, six experimental groups (two resin overlays with different layer combinations and three LCUs) were tested with seven DCRC specimens each (n=7). The VHN and the SBS of the DCRC to bovine dentin were analyzed with two-way analysis of variance (ANOVA) at a significance level of 5%, followed by post-hoc comparisons with Duncan test.

RESULTS

Surface Hardness of DCRC Light Cured Through Resin Overlays

Two-way ANOVA showed a significant effect for the main factors (layer thickness, $p<0.001$; LCU, $p<0.001$) and for their interaction ($p<0.001$, Table 1). A higher VHN was observed when the DCRC was light cured through the 0.2-0.5-0.8 combination than when the DCRC was light cured through the 0.5-0.5-0.5 combination. The VHN of the DCRC was highest when light cured with S10, followed by FL2 and 360 (Table 2). The hardness ratio of upper surface hardness of the DCRC light cured through composite resin overlays relative to the upper surface hardness of the DCRC light cured directly is shown in Table 3. The ratio was 92.5% for the DCRC light cured with S10 through the 0.2-0.5-0.8 combination.

Table 2: *Mean (Standard Deviation) VHN of DCRC Light Cured Through Resin Overlays with Dentin-Enamel-Translucent Layer Combinations of 0.5-0.5-0.5 and 0.2-0.5-0.8[a]*

Layer Combination of Resin Overlay	LCU		
	360	FL2	S10
0.5-0.5-0.5	31.21 (0.65) Aa	36.29 (1.00) Ab	40.99 (0.94) Ac
0.2-0.5-0.8	32.67 (0.87) Ba	38.70 (0.62) Bb	45.41 (1.33) Bc
Without resin overlay	45.88 (0.95) Ca	48.19 (0.58) Cb	49.09 (0.58) Cc

[a] *Mean values followed by different capital letters (column) or small letters (row) are significantly different by Duncan test (p<0.05).*

Table 3: *Hardness Ratio of Upper Surface Hardness of DCRC Light Cured Through Resin Overlays Relative to Upper Surface Hardness of DCRC Light Cured Directly*

Layer Combination of Resin Overlay	LCU		
	360	FL2	S10
0.5-0.5-0.5	68.0	75.3	83.5
0.2-0.5-0.8	71.2	80.3	92.5

SBS of DCRC to Bovine Dentin Light Cured Through Resin Overlays

Two-way ANOVA showed a significant effect only for the main factors (layer thickness, $p=0.007$; LCU, $p<0.001$) and not for their interaction ($p=0.445$, Table 4). The DCRC light cured with S10 showed a higher SBS than the DCRCs light cured with FL2 and 360 (Table 5). The DCRC light cured with S10 through the 0.2-0.5-0.8 combination showed a significantly higher SBS than the DCRC light cured with S10 through the 0.5-0.5-0.5 combination (Figure 4).

DISCUSSION

The retention of resin inlays can be increased through photopolymerization of the DCRC in the internal wall of prepared tooth when there is insufficient time for chemical curing. In this study, the surface hardness and SBS tests were performed 10 minutes after the start of light curing because occlusal adjustment and finishing and polishing of the composite resin inlays are performed immediately after the light-curing procedure. Stress created by these procedures can have a detrimental effect on the quality of the bonding between the dentinal walls and the composite resin inlays.[25,26]

The results demonstrated that VHN and SBS of the DCRC to bovine dentin were dependent on the layer combinations of resin overlays and LCUs. A higher VHN was observed when the DCRC was light cured through the 0.2-0.5-0.8 combination than when the DCRC was light cured through the 0.5-0.5-0.5 combination. Also, a higher VHN was observed when the DCRC was light cured with S10 than when the DCRC was light cured with FL2 and 360, regardless of the layer combination of resin overlays. These results could be explained by the access of the curing light to the DCRC.

In our previous study with the same indirect composite resin,[17] the power density of 360, FL2, and S10 through the 0.5-0.5-0.5 combination was measured as 103 mW/cm^2, 239 mW/cm^2, and 347 mW/cm^2, respectively, whereas the power density of 360, FL2, and S10 through the 0.2-0.5-0.8 combination was measured as 141 mW/cm^2, 307 mW/cm^2, and 447 mW/cm^2, respectively. Therefore, the power density of LCUs through resin overlays could be enhanced by reducing the thickness of dentin layer while increasing the thickness of the translucent layer, since the dentin layer attenuated the curing light more than the enamel and translucent layers.[17] The VHN values from this study were in accordance with the power density of LCUs through both combinations of resin overlays.

Usually, the degree of conversion of a composite resin is assessed by the hardness[27,28] and the hardness ratio,[29] the ratio between the upper and lower surface hardness values of a 2-mm-thick composite resin specimen.[8,30] However, in this study, the hardness of the lower surface of the DCRC was not measured because the thickness of the DCRC was controlled to 1.0 mm. In clinical situations, the thickness of resin cement was reported to be less than 300 μm.[31] In our pilot study, however, hardness testing of a 300-μm-thick DCRC resulted in perforation of the resin cement specimens. Therefore, the thickness of the DCRC had to be increased to 1 mm.

According to previous studies, the relative hardness of a composite resin was used to determine the extent of polymerization. The surface hardness of a

Table 4: *Summary of Two-way ANOVA for Main Factors (Layer and LCU) and Their Interactions for SBS*

Source of Variation	Sum of Squares	*df*	Mean Square	F	Significance
Layer combination of resin overlay	7.451	1	7.451	8.319	.007
LCU	209.952	2	104.976	117.207	<.001
Layer combination of resin overlay * LCU	1.483	2	0.742	0.828	0.445
Error	32.243	36	0.896		
Total	3044.299	42			

Table 5: Mean (Standard Deviation) SBS (in MPa) of DCRC Light Cured Through Resin Overlays to Bovine Dentin[a]			
Layer Combination of Resin Overlay	**LCU**		
	360	**FL2**	**S10**
0.5-0.5-0.5	4.97 (0.86) Aa	8.35 (0.81) Ab	9.88 (0.88) Ac
0.2-0.5-0.8	5.30 (0.97) Aa	9.33 (1.01) Ab	11.09 (1.12) Bc

[a] *Mean values followed by different capital letters (column) or small letters (row) are significantly different by Duncan test ($p<0.05$).*

test group was compared with that of a control group with maximum polymerization. A ratio of 90% was suggested to be acceptable for a clinical situation.[32] In a study by Arrais and others,[25] the monomer conversion in auto- and dual-polymerizing modes of a DCRC was investigated and the ratio of conversion in auto-polymerized relative to dual-polymerized modes was calculated and termed the "potential of cure." This concept was applied to our study, and the ratio of upper surface hardness of the DCRC light cured through composite resin overlays relative to upper surface hardness of the DCRC light cured directly was calculated. The hardness ratio was more than 90% only when the DCRC was light cured with S10 through the 0.2-0.5-0.8 combination.

To simulate the clinical situation of resin inlay cementation with a DCRC, the SBS test was performed 10 minutes after the start of photopolymerization. The SBS of the DCRC light cured with S10 through the 0.2-0.5-0.8 combination was significantly higher than that of the DCRC light cured with S10 through the 0.5-0.5-0.5 combination. This is in accordance with the hardness ratio of the DCRC light cured through resin overlays and the control. The DCRC light cured with S10 through the 0.2-0.5-0.8 combination was the only group with a hardness ratio greater than 90%. Therefore, it can be assumed that only the DCRC light cured with S10 through resin overlays in a 0.2-0.5-0.8 combination showed proper early photopolymerization. The SBS of the DCRC light cured with 360 and FL2 through the 0.2-0.5-0.8 combination was higher than the SBS of the DCRCs light cured with 360 and FL2 through the 0.5-0.5-0.5 combination; however, the difference was not significant. It can be assumed that the LCUs with a lower power density resulted in insufficient photopolymerization of the DCRC through either combination. Therefore, LCUs with a higher power density should be recommended for photopolymerization of the DCRC under resin inlays, since there are limitations in increasing the amount of curing light through resin inlays with reduced dentin-layer thickness.

This study focused on light curing of the DCRC through resin inlays, and the possible shade alteration of the resin inlays due to decreased dentin layer thickness and increased translucent layer thickness was not taken into account. Therefore,

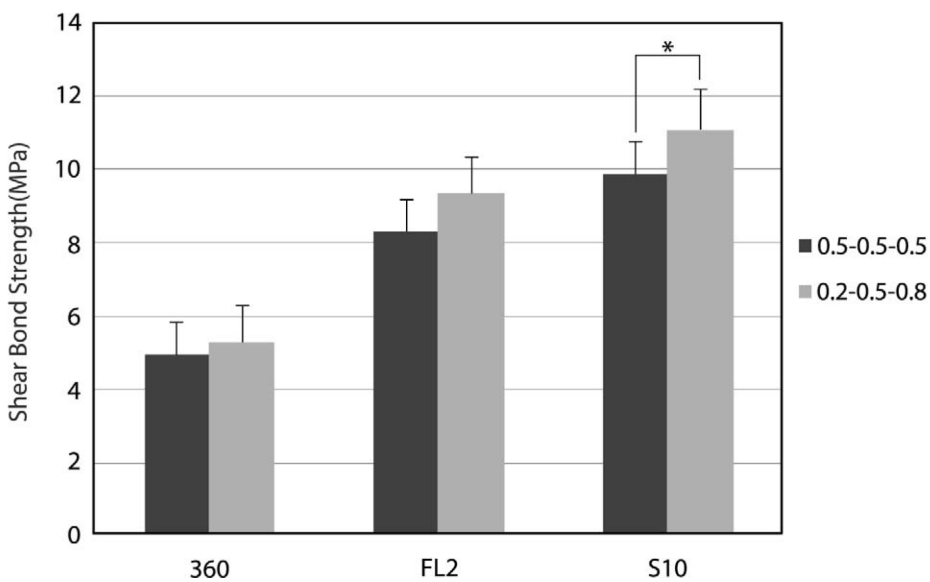

Figure 4. *SBSs (in MPa) of DCRC light cured through resin overlays to bovine dentin. Asterisk (*) indicates significant difference between groups ($p<0.05$).*

additional studies are needed to achieve maximal light curing of the DCRC through resin inlays with minimal shade alteration from the designated shade as the resin inlays are an esthetic treatment option.

CONCLUSIONS

Within the limitations of this study, the VHN and the SBS of the DCRC to bovine dentin under resin inlays could be increased by reducing the dentin-layer thickness while increasing the translucent-layer thickness. Also, LCUs with a higher power density are recommended for light curing the DCRC, since there are limitations in increasing the amount of curing light through resin inlays with reduced dentin-layer thickness.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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