

# Effect of Light-Emitting Diode (LED) Curing Modes on Resin/Dentin Bond Strength

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#### Abstract

**Purpose:** This study aimed to compare the effect of three curing modes of a high-powered curing-light source on the shear bond strength and marginal gap of light- and dual-cured adhesive resin cements to dentin.

**Materials and Methods:** Twelve freshly extracted intact human mandibular molars were selected for this study and stored in the saline solution. Three of the axial surfaces of the teeth were prepared to obtain flat dentinal surfaces. Thirty-six ceramic disks (4-mm diameter, 2- mm thick) were constructed from a pressable glass-ceramic (Vision). The discs were etched with hydrofluoric acid and primed, and then divided into two equal groups, groups I and II (n = 18 each). Two adhesive systems were used following manufacturer's instructions. The discs of group I were bonded to the conditioned dentin surface using adhesive resin (Rely X Veneer), and group II discs were bonded to dentin using Rely X ARC. For each group, the resin was cured using three modes (fast, ramp, pulse). Interfacial gap at the dentin/resin interface was measured at eight predetermined sites for each specimen using a stereomicroscope, and shear bond strength of the bonded specimens was carried out using a universal testing machine.

**Results:** Ramp-cured specimens recorded significantly higher mean shear bond strengths for both dual- and light-polymerized resins than those with fast and pulse modes. Moreover, fewer interfacial gaps were found at the resin/dentin interface in association with ramp cure modes of both resins. Most failures were adhesive failures at the dentin–resin luting agent (RLA) interface in specimens polymerized using high-powered LED fast or pulse modes, while a cohesive failure pattern within the resin was associated with the ramp-curing mode.

**Conclusion:** Within the limitations of this study, the shear bond strength of an RLA to dentin was found to be enhanced with light- or dual-polymerized adhesive resin using an LED light in ramp mode, whereas shear bond strength was significantly lower when polymerized using LED in fast or pulse modes.

Esthetic resin luting agents (RLAs) have become popular since their introduction, due to their high strength and esthetic appearance.<sup>1,2</sup> Increasing numbers of products are available on the market. These products are generally composite resins, provided with more than one polymerization mode to meet various clinical requirements. Autopolymerized luting agents are indicated for bonding metallic restorations, whereas lightpolymerized materials are used for laminate veneers, as light can penetrate through the restoration.<sup>3</sup> Dual-polymerized luting agents have been developed to combine autopolymerized and light-polymerized capabilities. The primary difference among activation modes is the initiation system. Autopolymerized luting agents are composed of two pastes, with the base paste containing aromatic tertiary amine and the catalyst paste containing benzoyl peroxide. The light-polymerized materials are a one-paste system using a photoactivator, such as camphorquinone, activated by blue light with a peak wavelength of approximately 470 nm. The reaction is accelerated by an organic amine (mostly aliphatic amine). The dual-polymerized luting agents have both initiation systems, so they are provided as two-paste systems as well, with the base paste containing camphorquinone, both aliphatic amine and aromatic tertiary amine, and the catalyst paste containing benzoyl peroxide.<sup>4,5</sup>

Success of all-ceramic restorations relies mainly on bonding to teeth using high-strength, low solubility, and thin-film adhesives. Consequently, light-polymerized RLAs are commonly used due to their improved physical and handling properties,<sup>6</sup> long working time, ease of excess removal, and color stability compared to dual- or self-cure systems.<sup>7</sup> Although photopolymerized materials have been used in general clinical practice, several problems have been identified. One particular problem associated with the photoactivation system is the lack of uniformity in the resin matrix conversion. The degree of conversion is related to polymerization contraction stresses. which develop much faster than with the other curing methods.8 A number of alternative polymerizing procedures have consequently been proposed to overcome insufficient material monomer conversion. Longer exposure is often used to improve properties of the materials.<sup>9-11</sup> Application of a high-intensity light source is another approach to overcome this insufficient conversion.<sup>12-14</sup> Several conflicting variables render identifying ideal light-polymerization standards difficult. Recently, it was observed that irradiation times used by dentists for lightpolymerizing cements were too short.<sup>15</sup> Longer polymerization times are mandatory to offset the decrease in light intensity incident upon the resin adhesive due to both the overlying material thickness and light source factors to achieve an adequate degree of polymerization.

The dimensional stability of composites is affected by the polymerization reaction of the matrix phase. This reaction produces gelation, in which the material is transformed from a viscous-plastic phase into a rigid-elastic phase. During polymerization of a composite, strains are produced by the forces that arise from polymerization shrinkage and are transferred to the tooth/restoration interface.<sup>16</sup> These strains remain built up in the restoration. If sufficient elastic compliance is not provided, the pre-stressed status of the restoration may cause fatigue within the material or fracture in some part of the restoration-bonding agent/tooth complex to compensate for the reduced volume. The amount of shrinkage is influenced by material properties such as resin composition, number of covalent bonds established between monomers, monomer size, filler type and content, stiffness, and application technique (including the volume of polymerized material, mode and velocity of polymerization, and direction of light application).<sup>16-18</sup>

Although halogen light units are used almost exclusively in the light-polymerization of RLAs, this technology has several drawbacks. Halogen bulbs have a limited effective lifetime of approximately 100 hours. They also produce large quantities of heat during polymerization cycles, which eventually results in the degradation of bulbs, reflectors, filters, and in turn, reduces polymerization capacity.<sup>19-21</sup> To overcome the problems inherent in halogen light-polymerization units, solid-state lightemitting diode (LED) technology has been proposed for lightpolymerization of dental materials.<sup>21-23</sup> An LED unit offers the advantages of minimal heat generation, as well as the capacity to operate for thousands of hours without a significant reduction in light output.<sup>19-21</sup> These units have a narrow spectral range with a peak around 470 nm, which matches the optimum absorption wavelength for the activation of the camphoroquinone photoinitiator present in light-activated dental resins. As a result, LED polymerization units do not require filters. Moreover, cordless LED units that provide reliable power and spectral output and do not require a cooling fan are available.<sup>24,25</sup>

Recently, manufacturers have turned their attention to highpowered (1000 mw/cm<sup>2</sup>) LED light units for the polymerization of dental resins; however, rapid polymerization may result in the formation of short polymer chains, thus shortening the pre-gel phase, resulting in the material's inability to adequately absorb polymerization contraction stresses.<sup>25,26</sup> Higher light intensity can also result in higher contraction stresses during resin polymerization, which may contribute clinically to microleakage, marginal discoloration, and insufficient shear bond strength. To achieve smaller marginal gaps, increased marginal integrity, and reduced microleakage, an initial low-intensity light exposure followed by a final high-intensity light exposure was proposed.<sup>27</sup> Studies have demonstrated that soft-start polymerization techniques significantly reduce polymerization strains and marginal gaps and enhance marginal integrity by improving material properties.<sup>15,20,21</sup> Polymerization units with higher light intensities have potential applications in prosthodontics.<sup>28</sup> Decrease in total polymerization time required for luting allceramic restorations may be beneficial for both the clinician and patient; however, the authors could identify few reports in the literature on the effects of high-powered LED polymerization units on the polymerization of RLAs.<sup>21,24,26,28,29</sup> Thus, the purpose of this study was to evaluate the effects of three curing modes of an LED polymerization unit (fast, ramp, pulse) on the marginal gap at the resin-dentin interface as well as the shear bond strength of light- and dual-polymerized RLAs to dentin.

## **Materials and methods**

Thirty-six ceramic specimens (4-mm diameter, 2-mm thick) were fabricated from heat-pressed leucite-reinforced glassceramic ingots (Vision, Wohlwend AG Dental, Schellenberg, Liechtenstein), shade A3. Wax pattern construction, spruing, investing, and burnout steps were performed according to the manufacturer's instructions. A special furnace (CeramPress Q50 Pressing furnace, Dentsply International, York, PA) was used for ceramic pressing according to the following specifications: start temperature of 700°C, heating rate of 60°C/min, final temperature of 950°C under vacuum, and 4.5 to 5 bar pressure. The ceramic discs were separated from their sprues using a sharp diamond disk. Low-fusing enamel porcelain was added to obtain a final ceramic thickness of 2 mm; firing started at 450°C and ended at 770°C under vacuum, and then, an autoglaze cycle was carried out using the previous cycle, but without vacuum. The bonding surfaces of the ceramic specimens were etched with 9% buffered hydrofluoric acid (Ultradent products, Inc., South Jordan, UT) for 1 minute and ultrasonically cleaned in distilled water. A layer of silane coupling agent (Rely X Ceramic primer, 3M ESPE, St. Paul, MN) was then applied to the ceramic surfaces and allowed to dry for 5 seconds.

Twelve freshly extracted intact human mandibular molars were selected for this study and stored in the saline solution. Three of the axial surfaces of the teeth were prepared to obtain a flat dentinal axial surface. The teeth were embedded in specimen holders perpendicular to the horizontal plane using an autopolymerizing acrylic resin (Karnak acrylics, Karnak, Egypt); the resin was extended to 2 mm below the cementoenamel



Figure 1 Ceramic discs bonded to three axial surfaces.

junction (CEJ). Three axial surfaces of each molar were wetground using a diamond stone secured to a precision-milling machine, to prepare flat surfaces within the dentin to accommodate the pressed disc and ensure maximum contact with it. A stereomicroscope (Leica MZ 12, Leica Microsystems AG, Heerbrugg, Switzerland) at 25× magnification was used to examine the ground dentin surfaces to ensure that they were free from enamel remnants. Dentin surfaces were then finished and polished sequentially using wet 360- and 600-grit silicon carbide paper.<sup>31,34</sup> After polishing, the distance between the CEJ and the occlusal surface of each specimen was approximately 4 mm. The prepared specimens were divided into two groups (n = 18 each) according to the method of cement activation (light or dual activation). Each group was further subdivided into three subgroups (n = 6) according to the mode of light activation (fast, ramp, pulse modes). The dentin surface of each specimen was etched with phosphoric acid gel (3M Scotchbond Etching Gel, 3M ESPE) for 15 seconds, rinsed for 10 seconds, and gently air-dried as necessary for wet-bonding. An adhesive agent (Adper Single Bond 2, 3M ESPE) was applied to the dentin surface in two consecutive coats and light-polymerized for 10 seconds. Each conditioned axial surface was designated a number to orient the specimens and mode of curing, such that each assigned axial surface underwent one mode of curing.

Three ceramic specimens were bonded to dentin surfaces of each tooth of group I (Fig 1), with a light-initiated RLA (Rely X Veneer, 3M ESPE). The RLA was mixed and applied to the bonding surface of the ceramic restoration. A special cementation jig was machined to apply a cementation load (1 kg) and stabilize the ceramic specimen during its bonding to the dentin surface, without interfering with light activation. Excess RLA was removed with a probe. The specimens were polymerized using one of three light-polymerization modes (fast, ramp, pulse) using a high-powered LED light-polymerization unit (Boss Dental, Walnut Creek, CA) (Table 1). The distance of the light tips from the specimen was 1 mm. The polymerization times used for LED fast, LED ramp, and LED pulse modes were fixed, preset by the LED unit manufacturer at 10 seconds for both fast and pulse modes, and 20 seconds for the ramp mode. The LED unit's batteries were recharged according to manufacturer's recommendations and replaced in their chargers following polymerization of each specimen. Group II ceramic specimens were bonded to the dentin surface with a two-paste, dual-polymerized resin cement (Rely X ARC) using each of the formerly used light-polymerization modes after excess cement was removed.

The disc-tooth interface for each disc specimen was marked at eight equidistant points circumferentially and examined using a stereomicroscope (SEM Olympus S2-PT, Olympus, Japan) at  $30 \times$  magnification. Photos of the resin–dentin interface were captured by a digital camera (DP-10 Olympus PT 10) connected to the microscope. The photos were analyzed by Image Analysis Software Image J (1.34, NIH, Bethesda, MD). The site of interest was drawn at the interface between the resin and dentine with fixed dimensions and saved to be applied to all photos. The gap at the region of interest was color thresholded and was automatically calculated by the image analysis software. The results were given in terms of  $\mu$ m<sup>2</sup>.

For bond strength testing, all specimens were mounted on a material testing machine (Model LRX-plus Lloyd Instruments Ltd, Fareham, UK) with a load cell of 5 kN, and data were recorded using computer software (Nexygen-4.1, Lloyd Instruments). Specimens were secured to the lower fixed compartment of the testing machine. Shear bond strength was determined by compressive mode of force, which was applied at the dentin/ceramic interface using a chisel-shaped metallic load applicator attached to the upper movable compartment of the testing machine traveling at a crosshead speed of 0.5 mm/min until debonding.<sup>34</sup> Loads were recorded in N. The load at failure was divided by the bonding area to express the bond strengths in MPa. The mode of failure was inspected using a stereomicroscope (SEM Olympus S2-PT) at 30× magnification and ranked. Failure within the cement layer was considered cohesive, failure at the resin-dentin interface was considered adhesive, and a combined failure mode was considered if cement was present on both dentin and disc substrates.

Data were recorded as mean and standard deviation (SD) values. One-way ANOVA was used to compare mean shear bond strengths and gap areas of the three polymerization modes. Duncan's post-hoc test was used for pairwise comparison between

Table 1 Light-polymerization unit and light-polymerization modes

Unit	Mode	Output and total time (seconds)	Profile			
Boss Dental, USA*	Light-emitting diode (LED)	Fast (10) Ramp (20)	Continuous energy output power of 1200 mW/cm <sup>2</sup> for 10 seconds Output power is raised gradually from 0 mW/cm <sup>2</sup> to 1200 mW/cm <sup>2</sup> in 10 seconds, power remains at 1200 mW/cm <sup>2</sup> during the next 10 seconds.			
		Pulse (10)	10 successive 1-second flashes at full power (1200 mW/cm <sup>2</sup> ) pulse activation mode, with a rest period of 250 ms between flashes			

\*Light intensity claimed by the manufacturer.

 Table 2
 Mean bond strength values (MPa) of both resins and three curing modes

Resin groups	Light		Dual		
Polymerization	Mean	SD	Mean	SD	P-value
Fast	8.9 <sup>b</sup>	1.2	13.6 <sup>b</sup>	1.3	<0.001*
Ramp	14.3ª	1.7	19.4 <sup>a</sup>	1.7	<0.001*
Pulse	10.4 <sup>b</sup>	1.9	12 <sup>b</sup>	1.5	<0.001*
	0.001*		<0.001*		

\*Significant at  $p \le 0.05$ , means with different letters within column are statistically significantly different according to Duncan's test

the means when the ANOVA test was significant. Student's *t*-test was used to compare means of shear bond strength and gap areas of the two curing methods. The significance level was set at  $p \le 0.05$ . Statistical analysis was performed with SPSS 15.0<sup>®</sup> (SPSS, Inc., Chicago, IL) Statistical Package for Scientific Studies for Windows.

#### Results

#### Shear bond strength

Mean shear bond strength measurements of the ceramic specimens bonded to dentin using light- and dual-polymerized resin cements showed that the dual-polymerization method contributed to an increased magnitude of shear bond strength, which differed significantly from the light-polymerization method (Table 2). Under both polymerization methods, the ramp-curing mode exhibited the greatest mean shear bond strength values, which differed significantly from both fast and pulse curing modes.

#### Gap area (dentin-resin interface)

Mean marginal gap measurements at the dentin/resin interface showed that the dual-polymerization resin contributed to smaller interfacial gaps, which differed significantly from that of the light-cured resin. Regarding the light-polymerized resin, statistically significant differences were apparent between the curing modes: the ramp mode showed the smallest gaps, followed by the pulse- and then fast-curing modes. As for the dual-polymerization method, statistically significant differences were found among the different curing modes. The smallest gaps were recorded in association with the ramp mode,

Table 3 Mean gap areas  $(\mu m^2)$  of both resins with different curing modes

Curing	Ligh	t	Dual		
Polymerization	Mean	SD	Mean	SD	P-value
Fast	12232.4	2.3	6373.3	4.1	<0.001*
Ramp	2091.6	3.4	3314.6	3.9	<0.001*
Pulse	3362.2	2.2	7883	2.5	< 0.001*

\*Significant at  $p \le 0.05$ 

Two types of bond failures were observed in this study: adhesive failure at the dentin/resin interface was most frequently encountered with pulse and fast modes, whereas cohesive failure within the RLAs was most commonly seen with the rampcuring mode.

## Discussion

Clinical performance of RLAs depends on a number of factors that may be related to dentin topography (patient related), type of RLA, bonding technique, or polymerization method used. Conversion of cross-linked, polymeric systems may play a major role in determining the ultimate physical and mechanical properties of the material. An increase in cross-linking density has been associated with an increase in fracture resistance.<sup>19</sup> Due to the significant role of polymerization in determining the shear bond strength of RLAs, the present study examined two luting resins with different activating systems (light- and dual-cured) and three curing modes to determine the most appropriate polymerization parameter with respect to the conditions tested.

Knowledge of adhesion of luting resins to dentin is fundamental for the success of the tooth-resin bond. Some polymerization units produce different levels of power (different watt outputs) and thus emit light with intensities that increase as a function of time, allowing the generation and regulation of energy and polymerization reaction. Despite that, polymerization of composite with high-intensity light sources is universally recommended, as they have the potential to improve the immediate depth of cure and mechanical properties of luting resins.<sup>30–32</sup> The results of the present study suggest that shear bond strengths of specimens polymerized using LED in the ramp mode were significantly higher than those recorded for fast and pulse modes when using both light- or dual-cured resins

With sequential polymerization, as with the ramp mode, the rate of conversion from monomer to polymer is slowed to prolong the pre-gel state of the paste, thereby minimizing stress build-up caused by polymerization shrinkage. This supports the findings reported earlier by Jung et al<sup>33</sup> and Nalcaci et al<sup>34</sup> who declared that maximum physical properties of composite luting agents under ceramic restorations are reached when the conversion rate is as high as possible. Those can be controlled by decreasing the rate of polymerization, thus decreasing the C factor (ratio between bonded and unbonded areas). Hence, it seemed logical that fast and pulse activation modes produced increased shrinkage forces, resulting in incomplete RLA polymerization, which was observed as lower bond strength values, especially in association with the light-polymerization method. These findings are in agreement with Caughman et al.<sup>35</sup> It is also believed that, because specimens polymerized using the high-powered LED unit in the ramp mode for 20 seconds had significantly higher shear bond strengths than those polymerized under fast and pulse modes for 10 seconds, lengthening the exposure period would increase energy density, thus increasing bond strength. In view of the increase in energy density when the duration of constant exposure is extended, it is likely that shear bond strength of RLA to dentin may increase with the LED fast and pulse modes when the duration of constant exposure is increased.

The preparation of specimens for this study attempted to simulate clinical conditions at the dentin/resin/ceramic interfaces. The aim of cementing three discs to each tooth using each of the tested curing modes was to achieve consistent results, due to the morphologic uniformity of the dentin structure within each tooth, thus offering reliable findings. The interfacial gaps obtained were not correlated with earlier studies, owing to the differences in test methodologies compared with previous works, where no attempt was made to measure the interfacial gap using a stereomicroscope. Studies mainly focused on the magnitude of the bond strength of resin to dentin.<sup>27,31,34</sup>

No statistical correlation between the recorded interfacial gaps and the obtained shear bond strength values was attempted in the present study. In most instances, mean gap measurements recorded along the dentin/resin interface did not coincide with the mean shear bond strength values.

Adhesive failure at the dentin/resin interface was the most common failure observed in specimens using LED fast and pulse modes. This supports the recorded low bond strengths in LED fast and pulse modes when compared to the LED ramp modes, which failed cohesively within the luting resin, which was seen covering both dentinal as well as ceramic disc surfaces.

The results of the present study support the findings of earlier studies<sup>31,35,36</sup> on the effectiveness of the dual-polymerization method of resin curing compared with the light-polymerization method as shown from shear bond strength data. The dual-polymerized specimens showed a higher degree of conversion due to the two initiation systems: chemical and light. Due to the wide variety of RLAs (different monomer composition and polymerization types) and energy levels of polymerization systems available for use in dental restorations, additional studies are needed to evaluate different RLAs, LED light intensities, and polymerization exposure times.

# Conclusion

Within the limitations of this study, the shear bond strength of RLAs to ceramic and dentin was found to be greater when the cement is light- or dual-polymerized using a high-powered LED light in the ramp mode, whereas shear bond strength was significantly lower when the resin cement was light- or dualpolymerized using LED in fast or pulse modes. In addition, smaller gaps at the dentin/resin interface were found when the ramp-curing mode was used.

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