

Delayed Photoactivation of Dual-cure Composites: Effect on Cuspal Flexure, Depth-of-cure, and Mechanical Properties

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Clinical Relevance

Delaying two minutes before light-curing after placing dual-cure composites can take advantage of shrinkage stress reduction of the slower self-cure component before the more rapid photoactivation without impacting curing depth or mechanical properties.

SUMMARY

Objectives: This study tested whether delayed photoactivation could reduce shrinkage stresses in dual-cure composites and how it affected the depth-of-cure and mechanical properties.

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Methods and Materials: Two dual-cure composites (ACTIVA and Bulk EZ) were subjected to two polymerization protocols: photoactivation at 45 seconds (immediate) or 165 seconds (2 minutes delayed) after extrusion. Typodont premolars with standardized preparations were restored with the composites, and cuspal flexure caused by polymerization shrinkage was determined with three-dimensional scanning of the external tooth surfaces before restoration (baseline) and at 10 minutes and one hour after photoactivation. Bond integrity (intact interface) was verified with dye penetration. Depth-of-cure was determined by measuring Vickers hardness through the depth at 1-mm increments. Elastic modulus and maximum stress were determined by four-point bending tests (n=10). Results were analyzed with two- or three-way analysis of variance and pairwise comparisons (Bonferroni; $\alpha=0.05$).

Results: Delayed photoactivation significantly reduced cuspal flexure for both composites at 10 minutes and one hour ($p \leq 0.003$). Interface was >99% intact in every group. Depth-of-cure,

elastic modulus, and flexural strength were not significantly different between the immediate and delayed photoactivation ($p > 0.05$). The hardness of ACTIVA reduced significantly with depth ($p < 0.001$), whereas the hardness of Bulk EZ was constant throughout the depth ($p = 0.942$).

Conclusions: Delayed photoactivation of dual-cure restorative composites can reduce shrinkage stresses without negatively affecting the degree-of-cure or mechanical properties (elastic modulus and flexural strength).

INTRODUCTION

Composite resin materials shrink when they polymerize, which can result in marginal failure, postoperative sensitivity, microleakage, secondary caries, and even tooth fracture.¹ In his landmark article that started the revolution of dental composites, Bowen pointed out that polymerization shrinkage would exert a force that could pull the material away from the cavity walls only after it reached a critical stiffness.² Before the composite reaches structural stiffness, contraction stress can still be relieved by viscous flow. The stress relieving flow can be extended by reducing the rate of polymerization, resulting in a decrease in shrinkage stress.^{3,4} This concept has been clinically applied by moderating the light intensity during photoactivation, as in the soft-start and pulse-delay photoactivation techniques.⁵⁻⁸

Slow polymerization has been a characteristic of self-cure composites because their polymerization process spans several minutes compared to the 20-40 seconds of most photoactivated composites.⁴ Reportedly, the cure speed of photoactivation could be up to 322 times faster than with self-curing.⁹ Recently dual-cure restorative composites were reintroduced that combine both curing mechanisms.¹⁰⁻¹² Dual-cure composites can presumably overcome curing depth limitations and thus become a candidate for bulk-fill procedures. Dual-cure composites may also allow further reduction in shrinkage stresses if the benefits of self-cure (low polymerization rate) and photoactivation (cure on demand) can be exploited using a delayed photoactivation technique. In this technique, photoactivation is delayed for a few minutes after the start of the mixing procedure to take advantage of the slow polymerization system of the self-cure component.^{13,14} This time delay has been shown to improve bonding of resin cements^{15,16} and reduction of shrinkage stress in core build-up materials and resin cements.^{13,14,17} However, reduc-

tion in shrinkage stress has not been tested yet for dual-cure restorative composites. Moreover, questions remain if delayed photoactivation can have negative effects on mechanical properties. The literature is not consistent on this concern.^{13,17,18}

The objective of this study was twofold: (1) determine the influence of delayed photoactivation of dual-cure restorative composites on polymerization shrinkage stresses and (2) evaluate the effect of delayed photoactivation on depth-of-cure and mechanical properties (elastic modulus and flexural strength). The hypotheses were that delayed photoactivation of dual-cure composites would reduce residual shrinkage stress and their mechanical properties.

METHODS AND MATERIALS

Restorative Composites and Curing Regimens

Two dual-cure composite materials—ACTIVA Bio-Active Restorative (Pulpdent Corporation, Watertown, MA, USA) and Bulk EZ (Danville Materials, Carlsbad, CA, USA)—were subjected to two curing regimens: 45 (immediate) or 165 seconds (with a 2-minute delay) after extrusion (mixing) of the composites. Material information is listed in Table 1.

Cuspal Flexure

Shrinkage stress conditions were assessed by measuring cuspal flexure of restored teeth. Typodont teeth were used for the cuspal flexure experiments. A previous study showed that cuspal flexure experiments with typodont teeth compared well with the cuspal flexure of natural teeth, replicating their stress distributions while standardizing the test conditions by precluding natural tooth variations.¹⁹ Forty typodont maxillary second premolars (Nissin-Kilgore, Nissin Dental Products, Inc, Kyoto, Japan) with prefabricated 3-mm-wide \times 3-mm-deep mesio-occlusal-distal (MOD) preparations were securely mounted in stainless steel rings (Figure 1A). All teeth were sandblasted with a MicroEtcher II Intraoral Sandblaster (Danville Materials, San Ramon, CA, USA) and Cojet Sand (3M Deutschland GmbH, Neuss, Germany) to provide adhesion. Scotchbond Universal adhesive (3M Deutschland GmbH) was applied to the cavity and rubbed for 20 seconds. A gentle stream of air was applied for about five seconds to evaporate the solvent. The adhesive was then light-cured for 10 seconds and followed by placement and 20-second light-cure of the dual-cured composites. The composites were either immediately light-cured (45 seconds after extrusion) or delayed light-cured (165 seconds after extrusion). All light

Table 1: Material Information			
Description	Material Information	Manufacturer	Composition
Bioactive dual-cure composite	ACTIVA BioActive-Restorative Shade A2 Lot 170126, 170326	Pulpdent Corporation	Patented, ionic-resin (Embrace resin), shock-absorbing resin component 56% by weight fillers (21.6% reactive ionomer glass fillers)
Dual-cure bulk fill composite	Bulk EZ Shade A2 Lot 47800, 123HU	Danville Materials	Patent-pending self-cure IntelliTek Technology
Typodont teeth Upper right second premolar	A25SAN-UR59M Lot 704808	Nissin Dental Product Inc	Bisphenol A diglycidyl ether epoxy resin 36-63 wt% Butyl glycidyl ether 4-8 wt% Inorganic Substance 29-52 wt% Colorant 0-8 wt%
Sandblast	3M ESPE CoJet Sand Lot 610969	3M Deutschland GmbH	Silicized sand, particle size 30 µm
Bonding agent	3M ESPE Scotchbond Universal Lot 619547	3M Deutschland GmbH	Bis-GMA, HEMA, dimethacrylates, ethanol, water, methacrylate functional copolymer of polyacrylic and polyitaconic acids, and photoinitiator 5 nm silane-treated spherical silica particles, 10% by weight
Sources: Product's Safety Data Sheet, Product Profiles, Product website, Product Technical Manual. Abbreviations: bis-EMA(6), bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, diurethane dimethacrylate.			

curing was carried out with an LED light-curing unit (Kerr, Orange, CA, USA) in standard curing mode with $1517 \pm 10 \text{ mW/cm}^2$ output according to the MARC Patient Simulator (BlueLight Analytics, Halifax, Nova Scotia, Canada). Sample size was 10 per group.

The samples were scanned (COMET xS Optical Scanner, Steinbichler Optotechnik GmbH, Neu-beuern, Germany) before composite placement (base-line), at 10 minutes and one hour after restoration. Tooth surfaces from the post-restoration scans were aligned with the baseline scan by precisely aligning

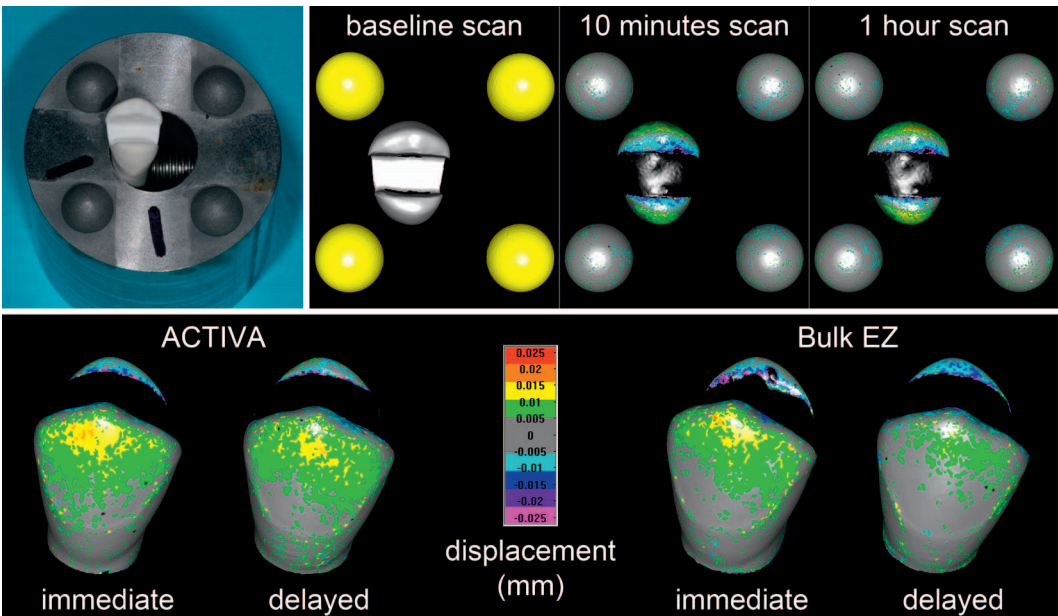


Figure 1. (A) Typodont tooth with MOD preparation fixed in a stainless steel mold with embedded reference spheres. (B) The 10-minute and one-hour restoration scans were precisely aligned with the preparation (baseline) scan using the reference spheres (yellow). Cuspal flexure was determined by calculating the difference between the restored tooth surfaces and the baseline. (C) Visualization of cuspal flexure of the four experimental groups using a color scale.

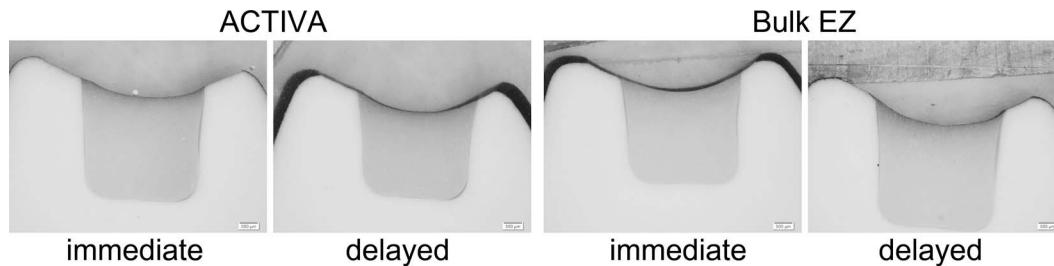


Figure 2. Samples from dye penetration experiment showing excellent bond integrity at the occlusal interfaces in every group.

reference spheres on the stainless steel rings using Cumulus software (copyright regents of the University of Minnesota) (Figure 1B). Cuspal flexure was calculated from the difference between the baseline and restoration scans for the buccal and lingual surfaces, calculated using custom developed software (CuspFlex).¹⁹ The effect of composite, regimen, and time was statistically analyzed using three-way analysis of variance (ANOVA; $\alpha=0.05$).

Bond Integrity

Intact bonding (tooth restoration interface integrity) was verified because it is important for the validity of the cuspal flexure results. Once all scans were completed, the restorations were finished using a 12-blade carbide bur to remove excess composite (flash covering occlusal margins) on the occlusal surface. The teeth were placed in 0.5 wt% basic fuchsin dye for 16 hours. The teeth were then sectioned buccolingually into 1-mm slides (Figure 2), and dye penetration along the occlusal interfaces was measured by two independent evaluators using a stereomicroscope with a CCD camera (SZX16 and UC30, Olympus, Tokyo, Japan). Percentage of intact bond was determined by dividing the length of the intact interface by the wall length (cavity depth). The results of both evaluators were averaged, or a consensus value was reached if the difference between the evaluators was more than 10%. The bond integrity results were statistically analyzed using two-way ANOVA ($\alpha=0.05$).

Depth-of-cure

Microhardness was used to assess the depth-of-cure. The composites were extruded in the rectangular slot (1.5 mm wide \times 2 mm high \times 8 mm long) of a plaster mold. The slot was covered with an orange glass plate (Bullseye Glass Company, Portland, OR, USA), while the end of the slot was covered with a thin clear glass coverslip (0.15 mm thick). The curing light tip was placed against the clear glass cover slip at the end of the covered slot to allow photoactivation

according to the two curing regimens. This configuration ensures that the curing light irradiates the composite from one direction.²⁰ Sample size was 10 per group.

After one hour, the glass covers were removed, and Vickers hardness was measured (QV-1000 Micro Hardness Tester, Qualitest USA LC, Fort Lauderdale, FL, USA) on the composite surfaces. This provided an indication of the degree of cure at various depths, from 0 to 6 mm. The hardness values were plotted along the depth to create depth-of-cure profiles. Statistical differences between curing regimens and depths were analyzed using two-way ANOVA and pairwise comparisons (Bonferroni; $\alpha=0.05$).

Elastic Modulus and Flexural Strength

Elastic modulus and flexural strength were measured to assess mechanical properties. Elastic modulus is the stiffness of the material, which determines its resistance to deformation and determines the stress values and distribution. Flexural strength represents the failure strength of the material.

The composites were extruded in the rectangular slot (2 mm deep \times 2.5 mm wide \times 25 mm long) of a vinyl polysiloxane mold. The filled mold was placed between two clear glass slides. Each composite sample was then polymerized, with photoactivation starting at 45 or 165 seconds after extrusion for the immediate or delayed photoactivation regimens, respectively. To ensure that the whole beam was light-cured, the tip of the curing light was moved along its length; both sides of the beam were light-cured (each side for a total of 80 seconds). Ten minutes after curing, the samples were removed from the mold and finished with 600-grit silicon carbide paper, after which the depth and width of each beam were measured with a digital caliper.

Beams were subjected to a four-point bending test one hour after cure in a universal testing machine (Instron 5567, Instron Corp, Norwood, MA, USA) at

Table 2: Cuspal Flexure, Bond Integrity, Elastic Modulus, and Flexural Strength (Mean \pm Standard Deviation)

Parameters	ACTIVA		Bulk EZ	
	Immediate	Delayed	Immediate	Delayed
Cuspal flexure (μm)				
10 minutes	9.4 \pm 3.1	8.1 \pm 3.0	7.8 \pm 2.6	5.5 \pm 3.1
One hour	12.4 \pm 3.1	10.9 \pm 2.5	10.4 \pm 2.5	7.7 \pm 2.7
Bond integrity (%)	100	100	99.66 \pm 0.72	99.96 \pm 0.13
Elastic modulus (GPa)	5.1 \pm 1.3	6.3 \pm 2.8	14.9 \pm 2.4	14.5 \pm 2.4
Flexural strength (MPa)	18.1 \pm 2.0	19.4 \pm 2.3	18.6 \pm 1.6	19.5 \pm 1.2

0.5-mm/min cross-head speed. Support span was 20 mm, with 10 mm between the two loading points. Displacement of the center of the beam during bending was measured using a deflectometer (Model 3540-004M-ST, Epsilon Technology Corp, Jackson, WY, USA). Each sample was loading until fracture. Load and displacement were recorded with Bluehill 2 software (Version 2.6, Instron Corp). Sample size was 10 per group.

The elastic modulus was calculated using $(11FL^3)/(64WH^3d)$, where F is the applied load, L is the distance between the two lower supports, W is the width of the sample, H is the height of the sample, and d is the displacement measured with the deflectometer. Flexural strength was calculated using $(3FL)/(4WH^2)$. The effect of composite and regimen was statistically analyzed using two-way ANOVA ($\alpha=0.05$).

RESULTS

The tooth cusps flexed inward during polymerization (Figure 1C). Cuspal flexure values of the two composites with immediate or delayed photoactivation measured at 10 minutes and one hour after restoration are shown in Table 2. The table shows that delayed photoactivation reduced cuspal flexure values, whereas cuspal flexure increased further between the 10-minute and one-hour measurement points. The effects of composite (ACTIVA versus Bulk EZ), curing regimen (immediate versus delayed photoactivation), and time (10 minutes versus 1 hour after polymerization) were all significant ($p \leq 0.003$).

The bucco-lingual sections of restored teeth after dye immersion showed hardly any dye leakage (Figure 2). Bond integrity (% intact bond) was higher than 99% in all groups (Table 2). No statistical differences were found for bond integrity between materials ($p=0.111$) or curing regimens ($p=0.206$).

Hardness gradually decreased with depth in the ACTIVA composite, whereas hardness remained constant through the depth for the Bulk EZ

composite, as shown in the hardness profiles in Figure 3. The statistical analysis confirmed that there was no significant difference in mean hardness values between immediate or delayed photoactivation for either composite ($p=0.605$ for ACTIVA, $p=0.425$ for Bulk EZ) and that there was no significant change in hardness throughout the 6-mm depth for Bulk EZ regardless of the curing regimen ($p=0.942$). For ACTIVA, hardness values decreased significantly until they bottomed out at about 4 mm depth ($p < 0.001$).

The elastic modulus and flexural strength values of the two composites after immediate or delayed photoactivation are also shown in Table 2. The

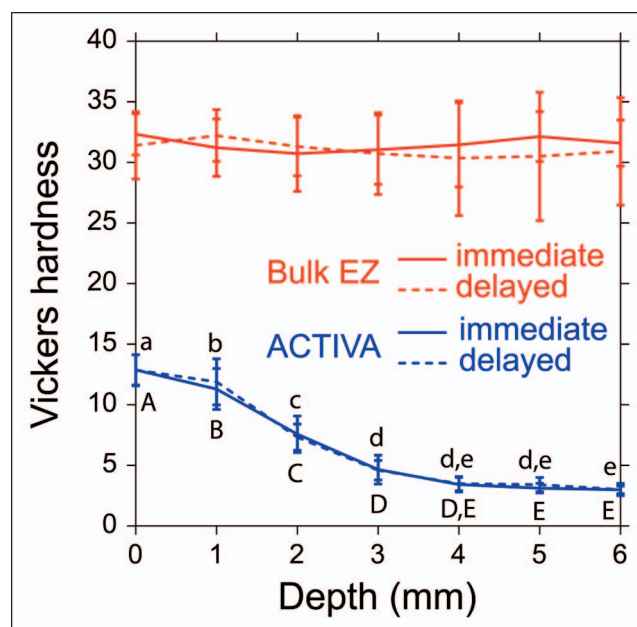


Figure 3. Depth-of-cure of ACTIVA and Bulk EZ composites with immediate or delayed photoactivation. There was no significant difference in hardness between immediate or delayed photoactivation. There was no significant change in hardness through the depth for Bulk EZ. For ACTIVA, same letters denote hardness values that are not significantly different (two-way ANOVA with pairwise comparison, $\alpha=0.05$; lowercase letters for immediate, uppercase letters for delayed).

elastic modulus of ACTIVA was significantly lower than Bulk EZ ($p < 0.001$), whereas their flexural strengths were not significantly different ($p = 0.597$). Immediate or delayed photoactivation had no significant effect on the elastic modulus ($p = 0.509$) or flexural strength ($p = 0.067$).

DISCUSSION

The first hypothesis that delayed photoactivation of dual-cure composite restorations reduces shrinkage stress was accepted because the delayed photoactivation regimen reduced cuspal flexure. Before further discussing this finding, three important observations about the testing method need to be made. Shrinkage stress was not measured directly (because stress cannot be measured directly), but cuspal flexure was used as an indication of stresses within the restored teeth.²¹ Second, typodont teeth with standardized preparation were used instead of extracted teeth to avoid natural variation in size, shape, and properties while still maintaining clinically relevant stress distributions.¹⁹ This decreased standard deviation and thus increased the resolution of the experiments, an important aspect considering how small the associated cuspal deformation is. Third, cuspal flexure values are only useful as stress analogies if the restorations are bonded. The required bond condition was confirmed with the close to 100% bond integrity. Note that the strength of the bond was irrelevant as long as bond integrity was maintained.

The reduction in shrinkage stress when photoactivation of dual-cure restorative composites was delayed is in agreement with other reports for dual-cure core build-up composites and dual-cure resin cements tested with delayed photoactivation.^{13,14,17} As discussed in the Introduction, the reduction in shrinkage stress can be attributed to a reduction in rate of polymerization when the photoactivation component is delayed. A slower polymerization rate is thought to increase pre-gel flow that relieves the accumulation of shrinkage stresses.^{5,22} Many studies have confirmed reduction in shrinkage stress in photoactivated composites if photoactivation regimens were altered to slow down the polymerization process,^{5,7,8,23} although there are also suggestions that the lower shrinkage stresses might be the result of compromised structural integrity.²⁴

Therefore, for our second hypothesis, we tested the possibility that delayed photoactivation compromised structural properties of the dual-cure composites. In the literature there have been various

outcomes for dual-cure materials. Some reported that dual-cured resin cements had higher cure with immediate photoactivation,¹⁸ whereas others argued that immediate light exposure could cause formation of polymer chains that interfere with the polymerizing process of the self-cure component by entrapping polymerization promoters.²⁵ Another study reported that elastic modulus and hardness of dual-cure resin cements were unaffected by a three- to five-minute delayed photoactivation.¹⁷ Our study found no significant differences in hardness profile, elastic modulus, or flexural strength between the immediately or delayed photoactivated dual-cure composites. The second hypothesis was therefore rejected as no evidence was found to suspect that delayed curing had altered or compromised structural integrity of the cured materials. Nevertheless, although our results do not show evidence of compromised integrity, it is still not inconceivable that slight changes in polymer structure were introduced, as others have reported in photoactivated resins.²⁶ The changes in polymer structure that were reported in the photoactivated resins did not directly affect mechanical properties, but they made the resins more susceptible to ethanol softening. Thus far, such changes appear mostly academic, as they have not been shown to affect clinical performance, which most likely could only show up as long-term effects. Future studies should test long-term mechanical performance of dual-cure composites using artificial aging, as well as leaching of unpolymerized monomers.

The original reason for considering delayed photoactivation was reduction of residual shrinkage stress. Although the negative effect of polymerization shrinkage stress on restoration longevity may be difficult to separate from other long-term challenges faced in the oral environment, shrinkage stress should still be regarded as an unfavorable side effect of resin composites, especially during the first weeks after placement when hygroscopic expansion and stress relaxation have not yet moderated its effects.²⁷ The ability to reduce shrinkage stress remains therefore a highly desirable option for dental practitioners. However, to adopt delayed photoactivation as a clinical technique, it should also be practical. Clinicians may wonder if it is worth waiting for two minutes before photoactivating. We believe that there are situations where shrinkage stress can be critical, such as in the gingival area of proximal restorations. Maybe two minutes waiting with the bulk-filling technique takes less time than placing two layers of incremental filling. Clinicians

can also limit the time impact by multitasking: for example, using a dual-cure composite to fill the proximal cavity and then filling and shaping the occlusal cavity using regular composite before photoactivating them at the same time. It should also be noted that both manufacturers recommend waiting before photoactivation; for Bulk EZ at least 60 seconds until the material solidifies,²⁸ and for ACTIVA 20-30 seconds to mitigate polymerization stress and exothermic reaction.²⁹

Few dual-cure dental composites are currently commercially available. The advertised advantage of dual-cure composites is their unlimited curing depth, allowing bulk-filling and application in areas where access to light curing is limited. One of the two dual-cure composites in this study (ACTIVA) did not show an unlimited curing depth (Figure 3). Such depth-dependent behavior was also reported in some dual-cure resin cements.^{20,30} Bulk EZ had constant hardness throughout the depth, suggesting a more efficient self-curing component. Compositions of both dual-cure composites tested are proprietary and can therefore not be used to explain the outcomes. Notable is that Bulk EZ has a patent pending for IntelliTek Technology for its self-cure component, said to control its shrinkage.³¹ Furthermore, in addition to photoactivation and self-curing chemistry, ACTIVA also has a self-cure glass ionomer reaction. In terms of mechanical properties, Bulk EZ had higher hardness and elastic modulus than ACTIVA, whereas the flexural strengths of both dual-cured composites were similar. The low elastic modulus of ACTIVA may in part be due to a patented rubberized-resin component, which the manufacturer claims resists wear, fracture, and chipping.³² The hardness values of ACTIVA were relatively low and were in the same range as sealant materials and lower than a filled sealant.³³ ACTIVA has been designed to be bioactive and capable of releasing calcium, phosphate, and fluoride to stimulate mineral apatite crystal formation at the material-tooth interface.²⁹

Dual-cure restorative composites have not reached the mainstream market. Our own observation is that the major dental manufacturers do not currently have dual-cure restorative composites in their product line. Perhaps the most popular product at the moment is ACTIVA, which may be for its bioactive trait rather than its dual cure.³⁴ Supplementary finding in this study regarding the depth-of-cure shows that Bulk EZ composite has a more effective self-curing component than ACTIVA and may thus be more reliable as a bulk filling material.

For both dual-cure restorative composites, clinicians should be aware that delayed photoactivation is a practical option—although it increases chair time—to reduce shrinkage stress without affecting the depth-of-cure and mechanical properties.

CONCLUSION

Delayed photoactivation of two dual-cure composites reduced shrinkage stresses without negatively impacting depth-of-cure and mechanical properties.

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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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