

Effect of Immediate or Delayed Light Activation on Curing Kinetics and Shrinkage Stress of Dual-Cure Resin Cements

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Clinical Relevance

Under clinical conditions, delayed light activation of dual-cured resin cements may be advantageous in order to reduce shrinkage stress.

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SUMMARY

Objectives: This study evaluated the effect of light activation (absence, immediate, or delayed) on conversion kinetics and polymerization stress of three commercial dual-cured resin cements (Enforce, RelyX ARC, and Panavia F). **Methods:** Degree of conversion (DC) was monitored for 30 minutes using real-time near-Fourier transform infrared spectroscopy. The cement was mixed, placed on the spectrometer sample holder, and light activated either immediately or after five minutes (delayed light activation). When no light activation was performed, the materials were protected from light exposure (control). DC was evaluated at five and 30 minutes postmixture. Maximum rates of polymerization ($R_{p_{max}}$) were obtained from the first derivative of the DC vs time curve. Polymerization stress was monitored for 30 minutes in 1-mm-thick specimens inserted between two cylinders attached to a uni-

versal testing machine. Data were submitted to analysis of variance/Tukey tests ($\alpha=0.05$). **Results:** Immediate light activation promoted the highest DC at five minutes. At 30 minutes, only RelyX ARC did not present a significant difference in DC between activation modes. Enforce and Panavia F presented higher Rp_{max} for immediate and delayed light-activation, respectively. RelyX ARC showed similar Rp_{max} for all activation modes. The absence of light activation resulted in the lowest stress followed by delayed light activation, while immediate light activation led to the highest values. RelyX ARC showed higher stress than Enforce, while the stress of Panavia F was similar to that of the others. **Conclusion:** Delayed light activation reduced the polymerization stress of the resin cements tested without jeopardizing DC.

INTRODUCTION

During post cementation, the exposed cervical margin is readily accessible to the curing light. However, a significant reduction in irradiance occurs as a result of the light scattering within the resin cement itself and shadowing produced by both tooth structure and the post.¹ In this situation, dual-cure resin cements are indicated, as they provide optimal polymerization, either in the presence or the absence of the curing light. Nevertheless, some dual-cured resin cements are dependent primarily on light activation, and therefore a somewhat lower degree of conversion is expected when exposed to low irradiance or when light from the curing unit is not available at all.²⁻⁶

Other studies found similar properties between dual-cure resin cements tested after light activation or in the self-cure mode.^{7,8} It has been hypothesized that light activation may negatively affect the self-curing mechanism. The rationale is that the rapid formation of a cross-linked polymer on exposure to light would lead to entrapment of the reactive species, including activators and initiators needed for the self-cure reaction.^{3,9-12} Thus, a delay period between cement mixing and light activation would increase total free-radical concentration, which would lead to a higher overall degree of conversion, ultimately improving the cement's mechanical properties.^{3,13}

Unfortunately, a higher degree of conversion is also related to an increase in polymerization shrinkage stress.¹⁴⁻¹⁶ If stress exceeds the bond strength between the dental substrate and the adhesive system, a contraction gap will be formed,¹⁷⁻²⁰

jeopardizing the longevity of the restoration. It has been demonstrated that lower polymerization rate^{21,22} and lower elastic modulus²²⁻²⁵ may help reduce polymerization stress. Therefore, it is important to determine the effect of delayed light activation on polymerization reaction rate and stress development in comparison with immediate light activation of these types of products.

The purpose of this study was to investigate the effect of immediate and delayed light activation on polymerization kinetics and shrinkage stress of three commercially available, dual-cured resin cements. For control purposes, the cements were also tested in the absence of light. It was hypothesized that delaying the light activation of dual cured resin cements 1) does not compromise the degree of conversion, 2) reduces the maximum rate of polymerization, and 3) reduces polymerization stress.

METHODS AND MATERIALS

Degree of Conversion and Maximum Polymerization Rate (Rp_{max})

Names and composition of the dual-cured resin cements tested are listed in Table 1. Degree of conversion was assessed using real-time near-Fourier transform infrared spectroscopy (Vertex 70, Bruker Optik GmbH, Ettlingen Germany). The spectrometer used an extended KBr beam splitter (Bruker Optik) and a InGaAs detector (Bruker Optik). Spectra (two per second) were obtained between 4000 cm^{-1} and 9840 cm^{-1} , with a 4-cm^{-1} resolution. For all resin cements, equal volumes of base and catalyst were mixed for 15 seconds and inserted into a 0.8-mm-thick circular silicone mold (with a 3.5-mm radius) placed on a glass slide. A second glass slide was placed on top of the mold, and the assembly was stabilized with clamps before positioned on the spectrometer sample holder.

The following polymerization scenarios were tested for all resin cements:

- *Immediate light activation:* The resin cements were immediately light activated, according to the manufacturers' recommended times: 20s (Panavia F), 30s (Enforce), and 40s (RelyX ARC).
- *Delayed light activation:* The luting agents were light activated 5 minutes after beginning the mixing procedure for the same time durations described above. This delay period was chosen on the basis that it allowed the resin cements to achieve more than 50% of their maximum degree of conversion in the self-cure mode (pilot study).

Table 1: *Materials Used*

Material	Manufacturer	Composition*	Shade	Filler Load (%) by Volume ^a
Enforce	Dentsply	Base: Bis-GMA, TEGDMA, CQ, EDAB, BHT and DHEPT	Translucent	66
	(Petrópolis, RJ, Brazil)	Catalyst: Bis-GMA, BHT, EDAB, TEGDMA and BPO		
RelyX ARC	3M ESPE	Paste A: Bis-GMA, TEGDMA, dimethacrylate polymer, CQ, amine	Transparent (A1)	67.5
	(St. Paul, MN, USA)	Paste B: Bis-GMA, TEGDMA, dimethacrylate polymer, BPO		
Panavia F	Kuraray Co.	Paste A: 10-MDP, hydrophobic and hydrophilic dimethacrylate, CQ, BPO	Light	78
	(Osaka, Japan)	Paste B: Hydrophobic and hydrophilic dimethacrylate, DHEPT, T-isopropyl benzenic sodium sulfinate		
ED Primer	Kuraray Co.	Primer A: HEMA, 10-MDP, NM-aminosalicylic acid, DHEPT, water		—
		Primer B: NM-aminosalicylic acid, T-isopropyl benzenic sodium sulfinate, DHEPT, water		

Abbreviations: Bis-GMA, bisphenol-A glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; CQ, camphorquinone; EDAB, ethyl 4-dimethylamine b; BHT, butylhydroxytoluene; DHEPT, N,N-di-(2-hydroxyethyl)-4-toluidine; BPO, benzoylperoxide; 10-MDP, 10-methacryloyloxydecyl dihydrogenphosphate; HEMA, hydroxyethyl methacrylate.

^a Information provided by the manufacturer.

- *Chemical activation:* No light activation was performed, and polymerization relied on self-activation only.

Panavia F was mixed with ED Primer before the evaluation, as the manufacturer states that the primer is essential for proper cement polymerization.¹⁵ For this material, one drop of each of the primer liquids A and B was mixed for 10 seconds and gently air-dried for 5 seconds to evaporate the solvent. Thereafter, 0.1 µL (1.4 mg) of this solution was dispensed onto a glass slab using a micropipette (model NPX2; Nichipet EX, Santa Clara, CA, USA) and mixed with the base and catalyst pastes (27 mg of each paste) of the dual-cure cement for 15 seconds. The volume of ED Primer added to the cement was based in a previous study.²⁶

All analyses were performed under controlled temperature ($25 \pm 1^\circ\text{C}$) and humidity ($60 \pm 5\%$) conditions. The output of the light-curing unit (Optilux 501; Demetron Kerr, Orange, CA) was periodically

checked using a handheld radiometer (Model 100; Demetron Kerr) and was determined to be near 800 mW/cm^2 . The tip of the light guide was placed at a 45-degree angle with the cement specimen, 1 mm distant from the glass slide, in order to not interfere with the infrared beam. For the immediate light-activated groups, the curing light was triggered after the first two spectra were collected, as those spectra were used as the unpolymerized reference. The materials were shielded from ambient light for the self-cure and delayed light-activation modes during all the experiment and in the first five minutes, respectively. Shielding was performed by keeping the material inside the closed sample compartment.

Spectra were obtained for 30 minutes. The area under the peak located at 6165 cm^{-1} corresponding to the first overtone of the C-H stretch²⁷ of the vinyl group was integrated with the use of software (Opus v.6; Bruker Optik). Degree of conversion was calculated by dividing the area of one spectrum

obtained at a given time by the average area of the first two spectra (ie, corresponding to the unpolymerized cement). Three replicates were obtained for each experimental condition. Degree of conversion values corresponding to five minutes and 30 minutes were recorded. The maximum polymerization rate ($RP_{\max}$) was calculated as the first derivative of the conversion vs time curve.²⁷

Polymerization Stress

Polymerization stress was measured using a universal testing machine (Instron 5565; Instron, Canton, MA, USA). The test was performed using clear poly(methyl methacrylate) (PMMA)²⁸ cylinders (6 mm in diameter and 13 or 28 mm high) as bonding substrates. One flat end of the short cylinders was polished using 600- to 1200-grit SiC abrasive paper in order to allow the highest possible light transmission during light activation. The other end of the short cylinders and both ends for the long ones were airborne particle abraded with 100 μ m aluminum oxide. Afterward, methyl methacrylate monomer (Jet Acrílico Autopolimerizante; Artigos Odontológicos Clássico, São Paulo, Brazil) was applied to improve the wettability of the PMMA surface. The surfaces were then covered with a layer of unfilled bonding resin (Scotchbond Multi-purpose Plus; 3M ESPE, St. Paul, MN, USA), which was then light cured for 20 seconds.

The 28-mm cylinder was clamped to the upper chuck, while the 13-mm cylinder was attached to a stainless-steel fixture clamped to the lower chuck. The resin cements were mixed in the same manner as described for polymerization kinetics evaluation and inserted between the PMMA cylinders, with the distance set to 1 mm. An extensometer (model 2630-101; Instron) was attached to the cylinders in order to keep specimen height constant during the test. Any approximation between the fixation points of the extensometer caused by resin cement shrinkage was immediately compensated for by controlled movement of the crosshead in the opposite direction, with accuracy of 0.1 μ m.

Resin cements used in the dual-cured mode were light activated with the same curing unit and for the same exposures described above. However, because light was shone through the clear PMMA rod, the irradiance effectively reaching the resin cement was 570 mW/cm² (as measured by a dental radiometer). The light guide was placed in contact with the polished end of the lower cylinder. Software (Bluehill 2; Instron) was used to monitor stress development. The test was conducted at room temperature

(25 $\pm$ 1°C), and data were recorded for 30 minutes from the time of resin cement placement. The value recorded by the load cell corresponded to the force necessary to keep specimen height constant, in opposition to the force exerted by composite shrinkage. The maximum nominal polymerization stress was calculated by dividing the maximum force value by the cross-sectional area of the cylinder. Five specimens were tested for each experimental condition. Stress vs time data were plotted, and Hill's 3 parameter nonlinear regressions were used for curve fitting (Sigmaplot 10.0; Systat Software Inc, San Jose, CA, USA).

Statistical Analysis

Degree of conversion, $RP_{\max}$, and polymerization stress data were each subjected to two-way analysis of variance (ANOVA; cement and curing method as main factors) using statistical software Sigmaplot 3.5 (Systat Software Inc, San Jose, CA, USA). Multiple pairwise comparisons were performed using Tukey post hoc test. In all cases, a preset global significance level of 5% was adopted.

RESULTS

Degree of Conversion and Maximum Polymerization Rate

Degree of conversion at five minutes (Table 2) showed a significant effect for the factor "activation mode" ($p=0.153$) and for the interaction between the factors ($p=0.002$) but not for the factor "resin cement" ($p=0.153$). For Enforce and Panavia F, immediate light activation resulted in statistically higher conversion values compared to delayed light activation and self-curing mode. RelyX ARC displayed

Table 2: Means ($\pm$ SD) of Percent Degree of Conversion at Five Minutes^a

Activation Mode	Resin Cements		
	Enforce	RelyX ARC	Panavia F
Immediate light activation	72.8 (3.4) Aab	59.7 (6.3) Ab	82.4 (4.1) Aa
Delayed light activation	37.6 (10.7) Ba	39.7 (13.1) Ba	28.0 (10.6) Ba
Self-cure	47.6 (3.5) Ba	44.0 (2.3) Ba	26.5 (4.8) Bb

^a Means followed by the same letters (uppercase, column; lowercase, row) are not statistically different ($p>0.05$).

similar conversion at five minutes among the three activation modes. However, cements ranked differently in each curing mode. With immediate light activation, DC of Panavia F reached the highest conversion, not significantly different from Enforce, while RelyX ARC was similar only to the latter. When light activation was delayed or in self-cure mode, conversion values for all three cements were not significantly different.

Conversion values obtained at 30 minutes are shown in Table 3. The factors “resin cement” ($p<0.001$) and “activation mode” ($p<0.001$) were significant, as was their interaction ($p=0.003$). For immediate light activation, Panavia F showed the highest values of DC, while RelyX ARC reached the lowest values. Conversion values for Enforce and RelyX ARC were not significantly different for delayed light activation and self-cure modes. Panavia F reached highest conversion when tested in delayed light-activation mode and similar values to other cements in self-cure mode. Comparison among cements revealed that Panavia F reached the highest conversion in both light-cure modes, while conversion values for Enforce and RelyX ARC were not significantly different. Conversion values 30 minutes postmix among the three cements using the self-cure mode were not significantly different.

For the maximum rate of polymerization (RP_{max}), the statistical analysis also showed significant effects for the factors “resin cement” ($p<0.001$) and “activation mode” ($p=0.002$) and for the interaction between the factors ($p<0.001$; Table 4). For Enforce, reaction rate was significantly higher for specimens immediately light activated, while delayed light-activation and self-curing values were not significantly different. The RP_{max} for RelyX ARC was not

Table 3: Means ($\pm$ SD) of Percent Degree of Conversion at 30 Minutes ^a			
Activation Mode	Resin Cements		
	Enforce	RelyX ARC	Panavia F
Immediate light activation	74.8 (2.2) Ab	65.4 (6.7) Ac	89.0 (2.4) Aa
Delayed light activation	72.5 (0.4) ABb	72.2 (4.9) Ab	93.4 (3.0) Aa
Self-cure	67.1 (1.0) Ba	66.4 (3.9) Aa	73.4 (3.5) Ba
^a Means followed by the same letters (uppercase, column; lowercase, row) are not statistically different ($p>0.05$).			

Table 4: Means ($\pm$ SD) of Maximum Rate of Polymerization, RP_{max} (percent conversion/s) ^a			
Activation Mode	Resin Cements		
	Enforce	RelyX ARC	Panavia F
Immediate light activation	9.7 (1.7) Ab	10.5 (1.6) Ab	21.0 (1.2) Ba
Delayed light activation	1.0 (0.1) Bc	9.2 (4.3) Ab	29.1 (3.1) Aa
Self-cure	0.8 (0.2) Bc	7.8 (1.5) Ab	21.0 (1.7) Ba
^a Means followed by the same letters (uppercase, column; lowercase, row) are not statistically different ($p>0.05$).			

significantly different among all activation modes. Panavia F showed higher RP_{max} for the delayed activation mode than using either of the other two modes. Comparison of RP_{max} among cements revealed that Panavia F had the highest value regardless of the curing method, followed by RelyX ARC and Enforce. No significant difference in RP_{max} was observed between the latter two cements when using immediate light activation.

Representative rate of polymerization profiles for the different polymerization scenarios are shown in Figures 1 and 2. For all resin cements, the peak of RP_{max} occurred within the first few seconds of the reaction when light activation was applied immediately. The peak in the polymerization rate curve was displaced to the right when the resin cements were only chemically activated. The delayed light activa-

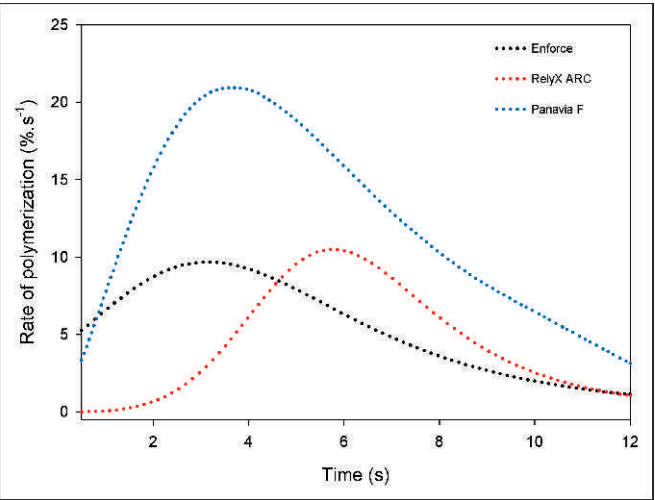


Figure 1. Representative polymerization rate profiles for the immediate light-activation mode.

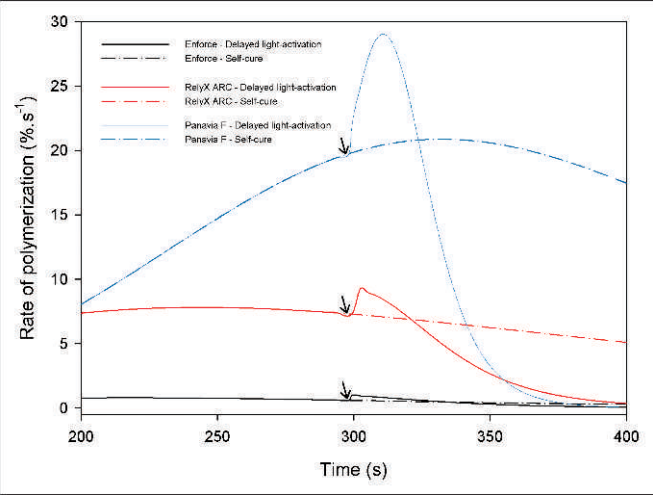


Figure 2. Representative polymerization rate profiles for the self-cured and delayed light-activated groups. Arrows indicate the moment when the light activation was performed.

tion generated a second peak during the autodeceleration of polymerization reactions for RelyX ARC and Enforce, while this second peak was observed during autoacceleration for Panavia F.

Polymerization Stress

ANOVA showed significant effect for the main factors on maximum polymerization stress values (resin cement: $p=0.024$; activation mode: $p<0.001$) but not for their interaction ($p=0.46$). Pairwise means comparisons are shown in Table 5. RelyX ARC presented higher maximum polymerization stress than Enforce. Panavia F showed intermediate values without significant differences from the other resin cements. For all three resin cements, the highest stress values were obtained with immediate light activation. The use of delayed light activation

resulted in intermediate stress values, while the lowest values were detected when no light activation was performed. A 19% reduction in stress was seen using the delayed mode compared to immediate light activation, while when used as self-curing materials, nominal stress values were four times lower than with immediate light activation.

The stress development profiles are illustrated in Figure 3. The coefficient of determination was greater than 0.9 for all curves. For the immediate light-activation mode, a fast increase in stress was observed in the first seconds. After this period, the slope of the curve decreased, reaching a plateau around 200 seconds for all cements. A gradual increase in stress was observed in the absence of light activation for all cements, reaching a plateau later (around 1200 seconds for Panavia F and RelyX ARC and 800 seconds for Enforce). A rapid increase in stress was observed at 300 seconds for the delayed light-activation mode, reaching a plateau around 900 seconds for all cements.

DISCUSSION

The results indicated that, among the three cements tested, only RelyX ARC reached similar conversion levels for all activation modes. Delayed light activation did not affect the degree of conversion after 30 minutes for any of the materials. Thus, the first hypothesis was confirmed. As expected, conversion values measured at five minutes were higher for the immediate light-activation and statistically similar for the delayed light-activation and self-cure groups. After 30 minutes, the statistically similar conversions for both immediate and delayed light-activated cements suggest that the possible increased mobility of the reaction media, caused by postponing light

Table 5: Means ($\pm$ SD) of Maximum Polymerization Stress (MPa) ^a				
Activation Mode	Resin Cement			Pooled Average
	Enforce	RelyX ARC	Panavia F	
Immediate light activation	4.7 (0.1)	4.9 (0.2)	4.9 (0.3)	4.8 (0.2) a
Delayed light activation	3.8 (0.2)	4.2 (0.6)	3.7 (0.3)	3.9 (0.5) b
Self-cure	0.9 (0.2)	1.4 (0.4)	1.2 (0.2)	1.2 (0.4) c
Pooled average	3.1 (1.7) B	3.5 (1.6) A	3.3 (1.6) AB	
^a For the pooled averages, means followed by the same letters are not statistically different ($p>0.05$).				

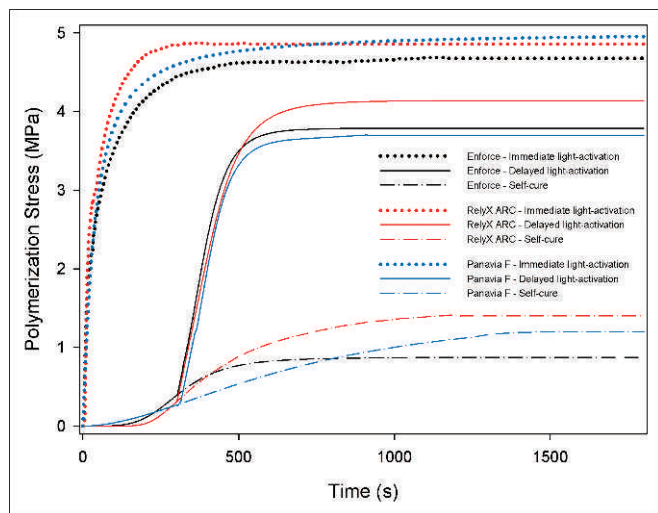


Figure 3. Representative polymerization stress development profiles.

activation, did not contribute to the extent of cure. Others also did not find differences between delayed and immediate light activation of one experimental dual-cured resin cement when a high-energy dose was used for light activation, as in the present study.³

The delay period before light activation significantly reduced the RP_{max} for Enforce to a value statistically similar to the reaction rate of the cements tested in self-cure mode. RelyX ARC showed statistically similar RP_{max} in all three curing modes, and Panavia F showed the highest values for delayed light activation. Thus, the second hypothesis tested was rejected. The increase in RP_{max} using the delayed light activation for Panavia F, different from the other cements, can be related to the lower-energy dose used for light activation (20 seconds), as previously demonstrated.³ A low RP_{max} indicates a slow polymerization reaction, which may help to reduce polymerization stress. During the polymerization reaction, viscosity and elastic modulus increase.^{9,10} Delaying the acquisition of a high elastic modulus allows the polymer chains to rearrange and accommodate the reduction of volume by plastic deformation.¹⁵ Furthermore, polymerization stress is related mainly to volumetric shrinkage and elastic modulus, both these being closely related to degree of conversion.¹⁵

Panavia F reached the highest values of RP_{max} and degree of conversion when the cements were light activated. However, polymerization stress of Panavia F was not different from that of the other cements. The inorganic content of resin composites has an important effect on elastic modulus.²⁴ High

filler content is associated with stiffer polymers and higher polymerization stress development.²⁵ Thus, it could be expected that Panavia F would present the highest stress, which did not occur. Monomer content also has an influence on stress. Less rigid monomers develop polymers with lower elastic moduli, which may also affect polymerization stress development.¹⁵ However, because the exact formulation of the evaluated cements is known, explaining the differences in stress among commercial materials based on their organic and inorganic contents is merely speculative.

Regardless of the differences detected for RP_{max} and degree of conversion, the self-cure mode yielded the lowest polymerization stress for all cements. Similarly, delayed light activation showed lower polymerization stress than immediate light-activation. Thus, the third hypothesis was accepted. As Enforce showed similar degrees of conversion between immediate and delayed light activation, differences in polymerization stress can be attributed to a reduced reaction rate. For Panavia F, the lower polymerization stress observed with self-cure mode can be credited to a lower degree of conversion. RelyX ARC showed similar values of DC and RP_{max} for all curing modes.

It is challenging to explain the differences in stress observed between immediate and delayed light activation, as conversion was similar and RP_{max} values were higher for the delayed light activation. In addition to RP_{max} and conversion, the polymer structure may also affect stress. Polymers with similar conversion may have different cross-link density.¹¹ Differences in polymer cross-link density influence elastic modulus, which also affects polymerization stress.²² Therefore, it is only possible to speculate that the delayed light activation could have altered the polymer structure in a way that elastic modulus would be lower, contributing to a reduced stress. The high degrees of conversion and RP_{max} values found with Panavia F may be explained by the fact that the cement was mixed with its primer, which made it less viscous, therefore allowing more carbon double-bond conversion prior to vitrification.

Clinically, dual-cure resin cements must be exposed to the curing light whenever possible. Light attenuation in clinical situations may be compensated for, to some extent, by increasing exposure time. Several studies have shown that marginal defects are frequently present in indirect restorations.¹⁸⁻²⁰ The results of this study showed strategies, such as delayed light activation, that may help reduce

polymerization stress and, ultimately, reduce marginal defects. The delay period used in this study was chosen because it is clinically feasible. However, only one delay period was evaluated, and the effect of other time periods should be evaluated. Another limitation of this study was that shrinkage stress was determined using 1-mm-thick specimens, much thicker than cement layers obtained clinically. Furthermore, the 30% attenuation in light activation through the PMMA rod limits the correlation between the values of polymerization stress and degree of conversion.

CONCLUSION

Based on the limitations imposed in the present study, the following conclusions may be drawn:

1. Delayed light-activation did not affect the degree of conversion of the resin cements. Only RelyX ARC reached similar conversion in either the presence or the absence of light, while Panavia F presented the highest conversion values for all curing modes.
2. The maximum rate of polymerization was affected by the activation mode, except for RelyX ARC. Enforce presented a higher rate when immediate light activation was performed, while Panavia F showed higher values for the delayed light activation.
3. Polymerization stress was significantly affected by the activation mode for all resin cements. Immediate light activation generated the highest values, and the lowest polymerization stress was obtained when no light activation was performed.

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