Delayed Photo-activation Effects on Mechanical Properties of Dual Cured Resin Cements and Finite Element Analysis of Shrinkage Stresses in Teeth Restored With Ceramic Inlays

CJ Soares • AA Bicalho • C Verissimo PBF Soares • D Tantbirojn • A Versluis

Clinical Relevance

Clinicians should take into account the mechanical properties when selecting resin cements. Slowing down the polymerization reaction allows more viscous flow with no effect on mechanical properties. Delayed photo-activation may be beneficial for cementation of indirect restorations in vital teeth.

SUMMARY

Objective: The aim of this study was to investigate the effect of delayed photo-activation on elastic modulus, Knoop hardness, and post-gel shrinkage of dual cure resin cements and how this affects residual shrinkage stresses in posterior teeth restored with ceramic inlays.

Methods and Materials: Four self-adhesive (RelyX Unicem, 3M ESPE; GCem, GC; Mono-

*Carlos J. Soares, DDS, MS, PhD, Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlandia, Minas Gerais, Brazil

Aline A. Bicalho, DDS, MS, PhD student, Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlandia, Minas Gerais, Brazil

Crisnicaw Verissimo, DDS, MS, PhD, Operative Dentistry and Dental Materials, School of Dentistry, Federal University of Uberlândia, Uberlandia, Minas Gerais, Brazil

Priscilla B. F. Soares, DDS, MS, Periodontology and Implantology, School of Dentistry, Federal University of Uberlândia, Uberlandia, Minas Gerais, Brazil

Cem, Shofu; and seT, SDI) and two conventional (RelyX ARC, 3M ESPE; and AllCem, FGM) dual cure resin cements for cementing posterior ceramic inlays were tested. Strain gauge and indentation tests were used to measure the post-gel shrinkage (Shr), elastic modulus (E), and Knoop hardness (KHN) when photoactivated immediately and 3 and 5 minutes after placement (n=10). Shr, E, and KHN results were analyzed using two-way analysis of variance followed by Tukey honestly significant difference post hoc tests (α =0.05). The

Daranee Tantbirojn, DDS, MS, PhD, Restorative Dentistry, University of Tennessee Health Science Center, Memphis, Tennessee, USA

Antheunis Versluis, PhD, Bioscience Research, University of Tennessee Health Science Center, Memphis, Tennessee, USA

*Corresponding author: Av. Republica do Piratini, S/No, Campus Umuarama, Bloco 4LA, Sala 4LA32, 38405-325; e-mail: carlosjsoares@umuarama.ufu.br.

DOI: 10.2341/15-090-L

experimentally determined properties were applied in a finite element analysis of a leucite ceramic inlay (Empress CAD, Ivoclar Vivadent) cemented in a premolar. Modified von Mises stresses were evaluated at the occlusal margins and cavity floor.

Results: Shr, E, and KHN varied significantly among the resin cements (p<0.001). Highest overall Shr values were found for RelyX Unicem; GCem had the lowest. Increasing the photo-activation delay decreased Shr significantly. Delayed photo-activation had no effect on E (p=0.556) or KHN (p=0.927). RelyX Unicem had the highest E values; seT and MonoCem had the lowest E values. AllCem and RelyX Unicem had the highest KHN and MonoCem had the lowest KHN. Cements with high Shr and E values caused higher shrinkage stresses. Stresses decreased with delayed photo-activation for all cements.

Conclusions: KHN and E values varied among the different resin cements. Residual shrinkage stress levels decreased with increasing photo-activation delay with all resin cements.

INTRODUCTION

In recent years, esthetic restorations became more popular as an alternative to amalgam restorations with good clinical longevity. In teeth with large cavities, where direct composite restorations can be compromised, indirect techniques such as adhesive ceramic inlays or onlays have been indicated to improve marginal adaptation and proximal contact.^{2,3} Close to 90% of ceramic inlays and onlays survive after 12 years. Despite their good longevity, postoperative sensitivity, marginal fracture, secondary caries, marginal deterioration, and discoloration are frequently identified as the causes for failure of this restorative procedure. 1,3-5 These failure mechanisms have been associated with polymerization shrinkage of resin cements used for luting ceramic inlays and onlays.5-7

Dual cure resin cements are widely used for luting ceramic restorations because they combine desirable features of photo- and chemical-cured systems and have shown good clinical performance. ^{1,8,9} Manufacturers recommend clinicians to wait before light curing the cements, because a time delay between the cement mixing step and photo-activation may favor the ability of dual-curing resin cement to bond to dentin. ¹⁰⁻¹² Additionally, a delay in photo-activation of dual-cured resin cements may reduce poly-

merization shrinkage stress.¹³ No studies have assessed how time lapse between manipulation of resin cements and photo-activation affects mechanical properties and residual shrinkage stresses in teeth restored with ceramic inlays.

The purpose of this study was to test the effect of delayed photo-activation of resin cements on 1) their mechanical properties (elastic modulus and Knoop hardness) and post-gel shrinkage and 2) residual shrinkage stresses in a premolar restored with a ceramic inlay restoration. The null hypotheses were that there would be no difference in mechanical properties and residual shrinkage stress among the resin cements due to photo-activation timing.

METHODS AND MATERIALS

Post-gel Shrinkage Measurements

Linear post-gel shrinkage of resin cement was determined using the strain gauge method. 14 The materials used were six dual cure resin cements: four were self-adhesive: RelyX Unicem (3M ESPE, St Louis, MO, USA), GCem (GC, Tokyo, Japan), MonoCem (Shofu, Tokyo, Japan), ad seT (SDI, Melbourne, Australia), and two are conventional resin cements: AllCem (FGM, Joinville, SC, Brazil) and RelyX ARC (3M ESPE). Composition and manufacturer information are listed in Table 1. Resin cement samples were shaped into hemispheres and placed on top of a biaxial strain gauge (CEA-06-032WT-120, Measurements Group, Raleigh, NC, USA) that measured shrinkage strains in two perpendicular directions. A strain conditioner (2101A Series, Micro Measurements Group) converted electrical resistance changes in the strain gauge to voltage changes through a quarter-bridge circuit with an internal reference resistance. Resin cement was photo-activated for 40 seconds with the light tip of the light-curing unit (XL 3000, 3M ESPE) placed at 1-mm distant from the surface of the resin cement. The radiant exposure was 32 J/cm² (800 mW/cm² × 40 seconds) measured by halogen light radiometer (Demetron, Danbury, CT, USA). Three photo-activation timings were tested: 1) light curing immediately, 2) after 3 minutes, or 3) after 5 minutes. Strain development (post-gel shrinkage) during polymerization shrinkage was monitored for 10 minutes, starting from the beginning of photo-activation. Ten specimens were tested for each resin cement at each time of photo-activation. The shrinkage strain was determined as the average of the strains in both perpendicular directions. The shrinkage strain values at 10 minutes were converted to volume percent

Resin cement Wt% Vol%		Vol%	Composition	Manufacturer	
RelyX Unicem	70	50	Base paste: fiberglass, phosphoric acid esters methacrylate, triethylene glycol dimethacrylate, silica treated silane, sodium persulfate	3M ESPE	
			Catalyst paste: fiberglass, substitute dimethacrylate, silane treated silica, p-toluenesulfonate sodium, and calcium hydroxide		
MonoCem	60	46	Mono-, di- and multifunctional acrylate resins, dual-initiators, fillers	Shofu	
AllCem	68	57	Bis-GMA, Bis-EMA, TEGDMA, camphorquinone, initiators, stabilizers, microfillers of Ba-Al-silicate, silica nanoparticles microfiller and nanofillers	FGM Dental Products	
GCem	71	57	4-MET, phosphoric acid ester monomer, water, UDMA, dimethacrylate, silica powder, initiator, stabilizer, fluoro-alumino-silicate glass, initiator, pigment	GC	
seT	67	45	Methacrylate ester phosphoric acids, UDMA, photoinitiator, fluoride aluminum silicate glass and pyrogenic silica	SDI	
RelyX ARC	67	45	Paste A: silane treated ceramic, TEGDMA, Bis-GMA, silane-treated silica, functionalized dimethacrylate polymer; 2-benzotriazolyl-4-methylphenol, 4-(dimethylamino)-benzeneethanol. Paste B: silane-treated ceramic, TEGDMA, Bis-GMA, silane-treated silica, functionalized dimethacrylate polymer	3M ESPE	

Abbreviations: BisEMA, bisphenol A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol A diglycidyl ether dimethacrylate; DMA, dimethacrylates; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MPS, 3-methacryloyloxypropyl trimethoxysilane; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

shrinkage by multiplying the strain values by 3% and 100%. Two-way analysis of variance (ANOVA) followed by Tukey honestly significant difference (HSD) post hoc tests (α =0.05) was used for the statistical analysis.

Elastic Modulus and Knoop Hardness Measurements

Five test specimens per resin cement were fabricated using a 2-mm-thick stone mold with a 5-mmdiameter circular opening. The stone mold was placed on a glass slide, filled with resin cement, and covered with another glass slide. The resin cement was light cured for 40 seconds (XL 2500, 3M ESPE) through the top glass slide at the three photoactivation times (immediately and after 3 or 5 minutes). Five Knoop indentations (MicroMet 5104, Buehler, Lake Bluff, IL, USA) were made to obtain an average Knoop hardness value for each specimen. The Knoop indentations were also used to determine the elastic modulus. 15-17 The decrease in the length of the indentation diagonals caused by elastic recovery of a material is related to the hardness/ elastic modulus ratio (H/E) according to the following empirical relationship: b'/a' = b/a - A (H/E), where b/a is the ratio of the diagonal dimensions a and b in the fully loaded state, given by a constant 0.140647. b'/a' is the ratio of the altered dimensions when fully recovered, and A = 0.45 is a proportionality constant. Two-way ANOVA followed by Tukey HSD post hoc tests (α =0.05) were used for the

statistical analysis of the parameters elastic modulus and Knoop hardness.

Residual Stress Calculation: Finite Element Analysis

To calculate residual stresses in a restored tooth, a two-dimensional (2D) finite element simulation was carried out for a leucite ceramic (Empress CAD, Ivoclar Vivadent, Schaan, Liechtenstein) inlay restoration with the cavity floor in dentin. The geometric model was based on a digitized buccolingual cross section of a premolar. Coordinates were obtained using ImageJ software (public domain, Java-based image processing and analysis software developed at The National Institutes of Health, Bethesda, MD, USA). Only the cervical portion of the root was simulated because the rest of the root did not affect the coronal stress distribution. 16 A simplified boundary condition was assumed at the cut-plane of the root (zero displacements in horizontal and vertical directions). The elastic modulus of enamel was 84 GPa and Poisson's ratio 0.30; the dentin elastic modulus was 18 GPa and the Poisson's ratio was 0.23. 18 The elastic modulus of leucite ceramic was 65 GPa and Poisson's ratio was 0.19.19 The shrinkage and elastic modulus values of the six resin cements were obtained from the experimental data. The Poisson's ratio was chosen to be the same for all resin cements at 0.35.19

The finite element analysis (FEA) was performed using MSC.Mentat (preprocessor and postprocessor)

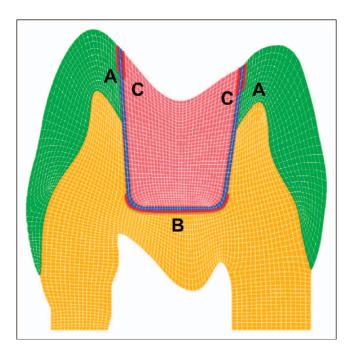


Figure 1. Mesh of digitized ceramic inlay restored premolar showing the locations where the stresses were recorded: (A) Enamel aspect of the restoration margin corresponding to with marginal gap formation. (B) Dentin aspect of pulpal floor corresponding to the potential area of post-operative sensitivity. (C) Ceramic aspect of the inlay margin corresponding to with ceramic marginal fracture.

and MSC.Marc (solver) software (MSC Software Corporation, Santa Ana, CA, USA). A plane strain condition was assumed for the tooth cross sections. Polymerization shrinkage was simulated by thermal analogy. Temperature was reduced by 1°C, while the linear post-gel shrinkage value was entered as the coefficient of thermal expansion. Modified von Mises equivalent stress was used to express the stress conditions, using compressive-tensile strength ratios of 3.3, 37.3, 3.0, and 3.9 for the ceramic, enamel, dentin, and resin cement, respectively. Enamel stress values were recorded in nodes along the interface at the occlusal margin (Figure 1A). Dentin stress values were recorded in nodes along the

interface at the pulpal floor (Figure 1B). Stress values in the ceramic aspect were recorded in nodes along the interface at the occlusal margin (Figure 1C). The mean values of the 5% highest stresses were determined.

RESULTS

Post-gel Shrinkage

The mean values and standard deviations for the volumetric post-gel shrinkage of six resin cements are presented in Table 2. Two-way ANOVA revealed statistical differences among the resin cements (p < 0.001) and timing of photo-activation (p < 0.001); however, no difference was found for interaction between resin cement and timing (p=0.319). The Tukey HSD test showed that the post-gel shrinkage values were progressively lower with delayed photo-activation, irrespective of resin cements. GCem had the lowest post-gel shrinkage values; seT had intermediate post-gel shrinkage, whereas RelyX Unicem, RelyX ARC, Monocem, and AllCem had high post-gel shrinkage values irrespective of photo-activation timing.

Elastic Modulus

The mean values and standard deviations for the elastic modulus of six resin cements are presented in Table 3. Two-way ANOVA revealed statistical differences among the resin cements (p<0.001); however, no difference was found for photo-activation timing (p=0.556) and for the interaction between resin cement and timing (p=0.061). RelyX Unicem had the highest elastic modulus values; GCem, RelyX ARC, and AllCem had intermediate values; and seT and MonoCem showed the lowest values, irrespective of photo-activation timing.

Knoop Hardness

The mean values and standard deviations for Knoop hardness of six resin cements are presented in Table

Resin cements	Post-gel shrinkage						
	Immediate	3 minutes	5 minutes	Pooled average			
RelyX ARC	0.97 (0.05)	0.73 (0.11)	0.64 (0.09)	0.78 (0.16) ^C			
AllCem	0.94 (0.06)	0.72 (0.08)	0.65 (0.06)	0.77 (0.14) ^C			
RelyX Unicem	0.93 (0.05)	0.82 (0.09)	0.77 (0.03)	0.84 (0.10) ^C			
MonoCem	0.91 (0.10)	0.83 (0.07)	0.71 (0.09)	0.82 (0.12) ^C			
seT	0.78 (0.13)	0.66 (0.08)	0.58 (0.13)	0.67 (0.16) ^B			
GCem	0.61 (0.03)	0.52 (0.06)	0.33 (0.02)	0.49 (0.17) ^A			
Pooled average	0.86 (0.15) ^c	0.71 (0.13) ^b	0.61 (0.17) ^a				

Mean values with same letters are not significantly different (p>0.05). Uppercase letters compare among resin cements and lowercase letters compare among time of photo-activation.

Resin cements	Elastic modulus					
	Immediate	3 minutes	5 minutes	Pooled average		
RelyX Unicem	18.6 (1.9)	18.4 (1.9)	18.1 (2.0)	18.4 (2.0) ^A		
GCem	14.0 (1.7)	13.7 (1.5)	13.4 (1.8)	13.7 (1.6) ^B		
RelyX ARC	12.3 (1.1)	12.7 (1.2)	12.8 (1.7)	12.6 (1.2) ^B		
AllCem	11.7 (1.3)	11.5 (0.9)	12.0 (1.1)	11.7 (1.1) ^B		
seT	9.5 (1.0)	9.6 (0.9)	9.7 (1.0)	9.6 (1.0) ^C		
MonoCem	9.0 (0.8)	9.1 (0.9)	9.7 (0.7)	9.2 (0.8) ^C		
Pooled average	12.5 (3.4) ^a	12.6 (3.2) ^a	12.5 (3.5) ^a			

Mean values with same letters are not significantly different (p>0.05). Uppercase letters compare between resin cements and lowercase letters compare among photo-activation timings.

4. Two-way ANOVA revealed statistical differences among the resin cements (p<0.001); however, no difference was found for time of photo-activation (p=0.927) or for the interaction between resin cement and timing (p=0.306). AllCem and RelyX Unicem had the highest mean Knoop hardness values; RelyX ARC, seT, and GCem had intermediate mean values, and MonoCem showed the lowest values, irrespective of photo-activation timing.

Residual Stress in FEA

Stress distributions are shown in Figures 2 (tooth structure) and 3 (ceramic inlay). They indicate stress concentrations (modified von Mises equivalent stress) in the tooth and inlay at the occlusal margins and in the dentin at the pulpal line angles. The highest levels of residual stresses were identified by calculating the mean values of the 5% highest modified von Mises stresses in the enamel aspect along the enamel/resin cement interfacial margin, the ceramic aspect along the ceramic/resin cement interfacial margin, and the dentin aspect along the pulp floor dentin/resin cement interface (Table 5).

The highest maximum stresses were found for the RelyX Unicem and the lowest for the seT. In all cases, the stress level decreased with increasing delays in photo-activation (Table 6).

DISCUSSION

Volumetric shrinkage is a consequence of the polymerization process, whereby the conversion of monomer molecules results in a cross-linked polymer network. During this polymerization reaction, resinous materials like cements change from a viscous to a predominantly solid substance, which can be characterized by the development of the elastic modulus. Residual shrinkage stresses are generated when the surrounding tooth structure restricts volumetric changes after the elastic modulus has developed. It can be theorized that this restriction is high for cementation of indirect restorations compared with direct composite resin restorations.

FEA was used to assess the stresses that polymerization shrinkage in the resin cement may pose on the tooth structure and ceramic inlay. This numer-

Resin cements	Knoop hardness					
	Immediate	3 minutes	5 minutes	Pooled average		
AllCem	51.0 (2.3)	50.5 (2.0)	50.7 (2.9)	50.7 (2.5) ^A		
RelyX Unicem	49.3 (4.3)	49.8 (4.6)	49.8 (3.9)	49.6 (2.5) ^A		
RelyX ARC	47.0 (4.0)	46.9 (3.2)	46.4 (3.5)	46.8 (3.5) ^B		
seT	45.6 (3.4)	45.8 (3.1)	46.2 (2.7)	45.9 (2.5) ^B		
GCem	44.0 (2.2)	44.8 (2.0)	43.6 (2.9)	44.1 (2.5) ^B		
MonoCem	31.8 (2.1)	30.6 (3.8)	30.8 (2,2)	31.1 (2.5) ^C		
Pooled average	44.8 (6.9) ^a	44.6 (6.8) ^a	44.8 (6.5) ^a			

Mean values with same letters are not significantly different (p>0.05). Uppercase letters are used to compare between resin cements and lowercase letters to compare among photo-activation timings.

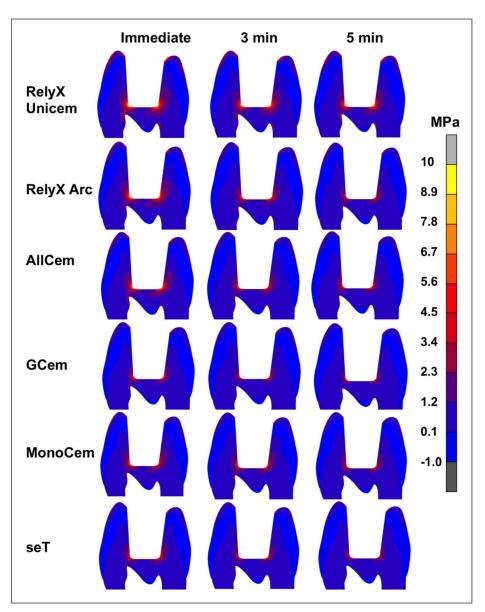


Figure 2. Residual shrinkage stress distributions (modified von Mises stress) in the tooth structure of a premolar restored with a ceramic inlay using six dual cure resin cements that were cured at three photo-activation timings (immediate and 3 or 5 minutes delayed).

Table 5: Mean and Standard Deviation of the Highest 5% Modified von Mises Equivalent Stress Values (MPa) Recorded in the Enamel and Ceramic Along the Occlusal Margins and in the Dentin at the Pulpal Floor

Resin cements	Enamel margins			Ceramic margins			Dentin, pulpal floor		
	Immediate	3 minutes	5 minutes	Immediate	3 minutes	5 minutes	Immediate	3 minutes	5 minutes
RelyX Unicem	25.4 (2.0)	22.2 (1.8)	20.9 (1.7)	26.1 (2.6)	22.8 (2.2)	21.4 (2.1)	29.1 (1.0)	25.4 (0.9)	23.8 (0.8)
RelyX ARC	17.9 (1.3)	13.9 (1.0)	12.3 (0.9)	18.4 (1.5)	14.3 (1.2)	12.6 (1.1)	21.0 (1.0)	16.3 (0.8)	14.3 (0.7)
AllCem	16.5 (1.2)	12.5 (0.9)	11.7 (0.8)	17.0 (1.4)	12.8 (1.0)	12.1 (1.0)	19.5 (1.0)	14.7 (0.8)	13.8 (0.7)
GCem	12.7 (1.0)	10.6 (0.8)	9.8 (0.7)	13.1 (1.1)	11.0 (0.9)	10.1 (0.9)	14.8 (0.3)	12.4 (0.5)	11.4 (0.5)
MonoCem	12.4 (0.8)	11.4 (0.8)	10.0 (0.7)	12.8 (0.9)	11.8 (0.8)	10.3 (0.8)	15.0 (1.0)	13.9 (0.9)	12.1 (0.8)
seT	11.4 (.08)	9.6 (0.7)	8.5 (0.6)	11.7 (0.9)	9.9 (0.7)	8.8 (0.7)	13.6 (0.8)	11.5 (0.7)	10.2 (0.6)

Table 6: Percentage Reduction in Stress Levels (Modified von Mises) of All Analyzed Regions When Photo-Activation Was Delayed 3 or 5 Minutes.

,							
Resin cements	Stress reduction						
	Immediate	3 minutes	5 minutes				
AllCem	_	-34%	-42%				
RelyX ARC	_	-29%	-46%				
seT	_	-19%	-34%				
GCem	_	-20%	-30%				
MonoCem	_	-18%	-24%				
RelyX Unicem	_	-15%	-22%				

ical method takes into account the geometrical restrictions experienced by the shrinking cement layer and combines them with the two major material properties involved in stress development: shrinkage and elastic modulus. Post-gel shrinkage was applied for the calculation because not all shrinkage induces stresses—only shrinkage that occurs when the resin cement has developed elastic properties. The elastic properties, which are the properties that prevent relief by viscous flow, were characterized by the elastic modulus. The determined stress distributions were therefore not determined by any individual property (shrinkage or

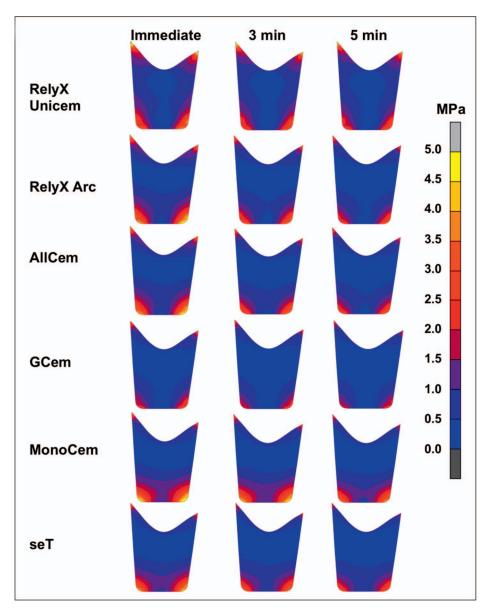


Figure 3. Residual shrinkage stress distributions (modified von Mises stress) in a ceramic inlay using six dual cure resin cements that were cured at three photo-activation timings (immediate, and 3 or 5 minutes delayed).

elastic modulus) or cavity configuration (bonding area), but were the result of the combination of all elastic properties and restriction factors (bonding and stiffness of tooth and inlay structures). However, by standardizing the restriction factors in our analysis, the differences between residual shrinkage stresses depended only on the shrinkage and elastic modulus of the resin cements.

The highest post-gel shrinkage values were found for RelyX ARC, AllCem, RelyX Unicem, and Mono-Cem. RelyX Unicem also had the highest elastic modulus, followed by GCem and RelyX ARC, The highest residual shrinkage stress level was found in RelyX Unicem, followed by RelyX ARC and AllCem. GCem, which had the lowest post-gel shrinkage but a relatively high elastic modulus, and MonoCem, which had the lowest elastic modulus but high postgel shrinkage, developed intermediate shrinkage stress levels, whereas seT with relative low post-gel shrinkage and elastic modulus values resulted in the lowest shrinkage stress. This confirms that, all other conditions being equal, it is the combination of postgel shrinkage and elastic modulus that determines the shrinkage stress levels.

Given full polymerization, mechanical properties such as elastic modulus and hardness were unaffected by the delayed photo-activation. However, delayed polymerization is advantageous by reducing the post-gel shrinkage values and thus decreased residual shrinkage stress levels in the restored tooth complex. Similar behavior has been shown previously for restorative composites, where photo-activation at lower light intensities reduced post-gel shrinkage. ^{24,25} This was explained by the slower reaction that allowed more viscous flow during polymerization and thus reduces the postgel portion of the total shrinkage.²⁶ Delaying the photo-activation of dual-cure resin cements used with endodontic posts has shown a similar effect, 13 slowing down the polymerization reaction, allowing more viscous flow and therefore reducing the postgel shrinkage without jeopardizing other mechanical properties as evidenced by the elastic modulus and Knoop hardness values that were unaffected by the delayed curing.

The decrease in post-gel shrinkage due to delayed photo-activation resulted in shrinkage stress reductions of 15%-34% with the 3-minute delays and 22%-42% with the 5-minute delays, depending on the resin cement type. Conventional resin cements (AllCem and RelyX ARC) showed higher reduction than self-etching resin cements (GCem, MonoCem, seT, and RelyX Unicem), probably because the

monomer composition of these cements is more reactive immediately after mixing. It has been suggested that photo-activation immediately after resin cement mixing may negatively affect the self-curing mechanism, because rapid formation of a cross-linked polymer on light exposure leads to entrapment of the activators and initiators needed for the self-cure reaction.²⁷ In our study, delayed photo-activation did not negatively affect the mechanical properties (Knoop hardness and elastic modulus), which is consistent with previous reports.^{28,29}

Failure of indirect restorations is often attributed to marginal fracture, marginal deterioration, discoloration, and secondary caries.^{3,4} Cement close to the restoration margin has been shown to have high stress concentrations, and this coincides with the area where bond and ceramic failure initiates. We also found residual shrinkage stress concentrations at the enamel and ceramic/resin cement interface close to the margin. Functional stresses in these areas can be elevated by these residual stresses concentrations, raising the overall stress levels at the occlusal margins and potentially causing bond failure, gap formation, or initiating cracks in the ceramic restoration. Gaps retain debris and pigments, resulting in marginal discoloration. Marginal discoloration is a frequent reason for replacement of indirect ceramic restorations because it can be misinterpreted as secondary caries. The cavosurface angle is also the thinner and thus weaker part of ceramic inlay and onlay restorations. Stress concentrations at this thin ceramic margin have been associated with formation of microcracks in ceramic restorations. 30,31 This study found residual shrinkage stress concentrations in the margins of the ceramic restoration, which may add an additional challenge to this vulnerable area. Avoiding or reducing shrinkage stresses at the margins could therefore increase the longevity of ceramic restorations. This study suggests that if clinicians delay photo-activation for 3-5 minutes after mixing the resin cements, the reduction in post-gel shrinkage will decrease shrinkage stresses and consequently lower the risk of marginal failure of indirect ceramic restorations. The 5-minute delay reduced post-gel shrinkage significantly more than the 3minute delay and further reduced the residual shrinkage stress. It should also be emphasized that the delayed photo-activation did not decrease the mechanical properties of the resin cement as evidenced by the elastic modulus and hardness values shown in Tables 3 and 4.

The main reason for using dual cure resin cement is that light cannot reach the floor of the cavity; therefore, the cure of the cement at the pulp floor depends on its auto cure component. Any accompanying shrinkage stress may cause postoperative sensitivity. Postoperative sensitivity is a frequent cause of indirect restoration failures.³² Confined polymerization shrinkage in tight interfacial spaces may cause fluid movement within the dentinal tubules and cause postoperative sensitivity. 33 Dentin deformation caused by shrinkage stress of the resin cements may also stimulate nerves directly or may exert mechanically induced dentinal fluid flow that triggers nerve activity.34 We found high stress concentrations at the pulpal floor for all resin cements. This may explain the relatively high percentage of postoperative sensitivity (13.3%) observed with dual cure conventional resin cement. 31 Although postoperative sensitivity tends to reduce with time, it is an undesirable incident. 35,36 Our study shows that delaying photo-activation may reduce shrinkage stresses at the pulpal floor and thus can reduce the incidence of postoperative sensitivity.

In this study, the inlay was simulated in a premolar only for simplification of the model because the findings can be extrapolated to clinically more common onlays and crowns. The results of in vitro studies should be carefully interpreted before being extrapolated to a clinical context. However, the general behavior of resin cements that are subjected to delayed photo-activation is consistent with previously reported observations and should also happen under clinical conditions. Considering no negative effect on the mechanical properties was found, this study suggests a potential strategy for clinicians to cement indirect ceramic restorations with delayed photo-activation. The current analysis indicates that the best results were found when photo-activation was delayed for 5 minutes after mixing the resin cements, which is clinically feasible considering the time used for seating the indirect restoration.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn: 1) Knoop hardness and elastic modulus values varied among the different resin cements; 2) post-gel shrinkage decreased significantly with increasing the photo-activation delay; 3) delayed photo-activation had no effect on elastic modulus and Knoop hardness of all resin cements; 4) resin cements with high post-gel shrinkage and elastic modulus values caused higher shrinkage stresses; 5) the residual stress is higher on the pulp floor than on

margins of the ceramic inlay; and 6) residual stresses on all regions analyzed decreased with 3 and 5 minutes of delayed photo-activation for all resin cements compared with immediate activation.

Acknowledgements

This project was funded by a post-doctoral grant from the CNPq- National Council for Scientific and Technological Development to Dr. Soares and a grant from FAPEMIG.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of Federal University of Uberlândia in Brazil.

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.

(Accepted 25 August 2015)

REFERENCES

- Beier US, Kapferer I, & Dumfahrt H (2012) Clinical longterm evaluation and failure characteristics of 1,335 allceramic restorations International Journal of Prosthodontics 25(4) 70-78.
- Soares CJ, Celiberto L, Dechichi P, Fonseca RB, & Martins LR (2005) Marginal integrity and microleakage of direct and indirect composite inlays: SEM and stereomicroscopic evaluation *Brazilian Oral Research* 19(4) 295-301.
- Lange RT, & Pfeiffer P (2009) Clinical evaluation of ceramic inlays compared to composite restorations Operative Dentistry 34(3) 263-272.
- Peumans M, Voet M, De Munck J, Van Landuyt K, Van Ende A, & Van Meerbeek B (2013) Four-year clinical evaluation of a self-adhesive luting agent for ceramic inlays Clinical Oral Investigation 17(3) 739-750.
- Batalha-Silva S, de Andrada MA, Maia HP, & Magne P (2013) Fatigue resistance and crack propensity of large MOD composite resin restorations: direct versus CAD/ CAM inlays Dental Materials 29(3) 324-331.
- Malhotra N, Kundabala M, & Shashirashmi A (2010) Strategies to overcome polymerization shrinkage—materials and techniques. A review *Dental Update* 37(2) 115-125.
- Feitosa SA, Corazza PH, Cesar PF, Bottino MA, & Valandro LF (2014) Pressable feldspathic inlays in premolars: effect of cementation strategy and mechanical cycling on the adhesive bond between dentin and restoration Journal of Adhesive Dentistry 16(2) 147-154.
- 8. Ozturk N, Usumez A, Usumez S, & Ozturk B (2005) Degree of conversion and surface hardness of resin

cement cured with different curing units *Quintessence International* **36(10)** 771-777.

- 9. Santos MJ, Mondelli RF, Navarro MF, Francischone CE, Rubo JH, & Santos GC Jr (2013) Clinical evaluation of ceramic inlays and onlays fabricated with two systems: five-year follow-up *Operative Dentistry* **38(1)** 3-11.
- Vrochari AD, Eliades G, Hellwig E, & Wrbas KT (2009)
 Curing efficiency of four self-etching, self-adhesive resin cements *Dental Materials* 25(9) 1104-1108.
- Faria-E-Silva AL, Peixoto AC, Borges MG, Menezes Mde S, & Moraes RR (2014) Immediate and delayed photoactivation of self-adhesive resin cements and retention of glass-fiber posts *Brazilian Oral Research* 28(1) 1-6.
- 12. Khoroushi M, Karvandi TM, & Sadeghi R (2012) Effect of prewarming and/or delayed light activation on resimmodified glass ionomer bond strength to tooth structures *Operative Dentistry* **37(1)** 54-62.
- Pereira RD, Bicalho AA, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2015) Effect of photo-activation timing on mechanical properties of resin cements and bond strength of fiberglass posts to root dentin *Operative Dentistry* 40(5) E206-E221.
- Sakaguchi RL, Sasik CT, Bunczak MA, & Douglas WH (1991) Strain gauge method for measuring polymerization contraction of composite restoratives *Journal of Dentistry* 19(5) 312-316
- Marshall DB, Noma T, & Evans AG (1982) A simple method for determining elastic-modulus-to-hardness ratios using Knoop indentation measurements *Journal of American Ceramic Society* 65 C175-C176.
- Soares CJ, Bicalho AA, Tantbirojn D, & Versluis A (2013) Polymerization shrinkage stresses in a premolar restored with different composite resins and different incremental techniques *Journal of Adhesive Dentistry* 15(4) 341-350.
- Bicalho AA, Tantbirojn D, Versluis A, & Soares CJ (2014) Effect of occlusal loading and mechanical properties of resin composite on stress generated in posterior restorations American Journal of Dentistry 27(3) 129-133.
- 18. Versluis A, & Versluis-Tantbirojn D (2011) Filling cavities or restoring teeth? *Journal of the Tennessee Dental Association* **91(2)** 36-42.
- Dejak B, Młotkowski A, & Langot C (2012) Threedimensional finite element analysis of molars with thinwalled prosthetic crowns made of various materials *Dental Materials* 28(4) 433-441.
- Davidson CL, de Gee AJ, & Feilzer A (1984) The competition between the composite-dentin bond strength and the polymerization contraction stress *Journal of Dental Research* 63(12) 1396-1399.
- 21. Versluis A, Douglas WH, Cross M, & Sakaguchi RL (1996) Does an incremental filling technique reduce polymerization shrinkage stresses? *Journal of Dental Research* **75(3)** 871-878.
- 22. Bicalho AA, Pereira RD, Zanatta RF, Franco SD, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—part I: cuspal

- deformation, bond strength, and physical properties *Operative Dentistry* **39(2)** E71-E82.
- Bicalho AA, Valdívia AD, Barreto BC, Tantbirojn D, Versluis A, & Soares CJ (2014) Incremental filling technique and composite material—part II: shrinkage and shrinkage stresses Operative Dentistry 39(2) E83-E92.
- Versluis A, Tantbirojn D, & Douglas WH (1998) Do dental composites always shrink toward the light? *Journal of Dental Research* 77(6) 1435-1445.
- Sakaguchi RL, & Berge HX (1998) Reduced light energy density decreases post-gel contraction while maintaining degree of conversion in composites *Journal of Dentistry* 26(8) 695-700.
- Versluis A, Tantbirojn D, & Douglas WH (2004) Distribution of transient properties during polymerization of a light-initiated restorative composite *Dental Materials* 20(6) 543-553.
- Rueggeberg FA, & Caughman WF (1993) The influence of light exposure on polymerization of dual-cure resin cements Operative Dentistry 18(2) 48-45.
- 28. Moraes RR, Faria-e-Silva AL, Ogliari FA, Correr-Sobrinho L, Demarco FF, & Piva E (2009) Impact of immediate and delayed light activation on self- polymerization of dual-cured dental resin luting agents *Acta Biomaterialia* **5(6)** 2095-2100.
- Faria-e-Silva A, Boaro L, Braga R, Piva E, Arias V, & Martins L (2011) Effect of immediate or delayed light activation on curing kinetics and shrinkage stress of dual-cure resin cements Operative Dentistry 36(2) 196-204.
- 30. Martin N, Jedinakiewick NM, Williams DF (1999) Cuspal deflection during polymerization of composite lutes *Journal of Dentistry* **27(1)** 29-36.
- 31. Pishevar L, Ghavam M, Pishevar A (2014) Stress analysis of two methods of ceramic inlay preparation by finite element *Indian Journal of Dental Research* **25(3)** 364-369.
- 32. Denner N, Heydecke G, Gerds T, & Strub JR (2007) Clinical comparison of postoperative sensitivity for an adhesive resin cement containing 4-META and a conventional glass-lonomer cement *International Journal of Prosthodontic* **20(1)** 73-78.
- Umer F, & Khan FR (2011) Postoperative sensitivity in Class V composite restorations: comparing soft start vs. constant curing modes of LED *Journal of Conservative Dentistry* 14(1) 76-79.
- 34. Linsuwanont P, Versluis A, Palamara JE, & Messer HH (2008) Thermal stimulation causes tooth deformation: a possible alternative to the hydrodynamic theory? *Archives of Oral Biology* **53(3)** 261-272.
- 35. Rosentiel SF, & Rashid RG (2003) Postcementation hypersensitivity: scientific data versus dentists' perceptions *Journal of Prosthodontic* **12(2)** 73-81
- 36. Blatz MB, Mante FK, Saleh N, Atlas AM, Mannan S, & Ozer F (2013) Postoperative tooth sensitivity with a new self-adhesive resin cement—a randomized clinical trial *Clinical Oral Investigation* **17(3)** 793-798.