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Effect of Photoactivation Timing on the Mechanical Properties of Resin Cements and Bond Strength of Fiberglass Post to Root Dentin

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Clinical Relevance

Delayed photoactivation may be beneficial for the clinical behavior of resin cements used to lute fiber posts. The root canal region is still a critical factor for the mechanical behavior of dual-cure resin cements.

SUMMARY

Objectives: This study tested the hypothesis that photoactivation timing and resin cement affect mechanical properties and bond strength of fiberglass posts to root dentin at different depths.

Methods: Fiberglass posts (Exacto, Angelus) were luted with RelyX Unicem (3M ESPE), Panavia F 2.0 (Kuraray), or RelyX ARC (3M ESPE) using three photoactivation timings: light curing immediately, after three minutes, or after five minutes. Push-out bonding strength, PBS (n=10) was measured on each root region (coronal, middle, apical). The elastic modulus (*E*) and Vickers hardness (VHN) of the cement layer along the root canal were determined using dynamic indentation (n=5). A strain-gauge test was used to measure post-gel shrinkage of each cement (n=10). Residual shrinkage stress was assessed with finite element analysis. Data were analyzed with two-way analysis of variance in a split-plot arrangement and a Tukey test (α=0.05). Multiple linear regression analysis was used to determine the influence of study factors.

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Results: The five-minute delay photoactivation timing significantly increased the PBS for all resin cements evaluated. The PBS decreased significantly from coronal to apical root canal regions. The mean values for *E* and VHN increased significantly with the delayed photoactivation for RelyX Unicem and decreased from coronal to apical root regions for all resin cements with the immediate-curing timing.

Conclusions: The PBS of fiber posts to root dentin, *E*, and VHN values were affected by the root canal region, photoactivation timing, and resin cement type. Shrinkage stress values decreased gradually with delayed photoactivation for all the cements.

INTRODUCTION

The esthetic and functional rehabilitation of endodontically treated teeth with less than two residual coronal walls usually requires the use of intraradicular posts.[1] Laboratory and clinical evaluations support the use of fiberglass posts for the retention of direct and indirect restorations.[2-6] Fiber posts have many advantages compared with metallic posts, especially the possibility of bonding to resin cements,[7] and have an elastic modulus (*E*) similar to dentin, which results in a lower stress concentration at the root, reducing the risk of root fractures.[4,6-8]

Several longitudinal assessments of fiber posts have shown a high success rate.[2,5,9-11] Most clinical failures were found to be related to debonding of the posts.[2,3,5,9-11] The fracture of the fiber post is also a commonly observed failure, mostly when used for restoring teeth without a ferrule.[10] The debonding occurred at the resin cement-dentin and/or resin cement-post interfaces.[12] Improving the adhesion at these interfaces will enhance the clinical performance of endodontically treated teeth restored with fiber posts.[13-16] The interface between resin cement and dentin is crucial for the retention of fiber posts.[15,17] The bonding at this interface can be influenced by several factors such as the root canal region, use of activators for conventional resin cements,[18] and properties and degree of conversion of the resin cements.[12,19-23]

Dual-cure resin cements have been widely used and were investigated in a number of clinical studies.[2,3,5] A chemical-cure is expected to adequately polymerize the cement in areas that cannot be entirely reached by a curing light, as well as in completely obscured regions. Despite the presence of some light, it may still not be enough for some dual-cure resin cements to reach an adequate degree of conversion.[12,20,21,23] Photoactivation has been recommended by manufacturers for different times after manipulation and insertion of the resin cement into the root canal.[22] Conventional dual-cure resin cements showed an increased degree of conversion after light curing, but the time elapsed from cement manipulation to light exposure had no influence on degree of conversion of the resin cement.[21,24] Self-adhesive resin cements were introduced in 2001 to simplify luting procedures and have been a good option to fix fiberglass posts due to their bonding performance and low viscosity.[25-28] No studies have assessed the influence of the time elapsed between the manipulation of resin cements and the light curing on the mechanical properties and bond strength of these cements for luting fiberglass posts.

Based on the bonding mechanism and the importance of pH-buffering for polymerization, a time delay between the cement mixing and photoactivation steps may favor the ability of dual-curing resin cement to bond to dentin.[29,30] A rapid increase of cement viscosity by light irradiation[31] may hinder the reaction of the acidic monomers with the dental tissues, which may affect the bonding mechanism.[30] Additionally, the delay of dual-cured resin cement photoactivation can reduce polymerization stress.[30] Another aspect that can affect the mechanical properties of resin cements and the bond strength of a post is the polymerization shrinkage of resin cements. Shrinkage of the cement can lead to considerable residual shrinkage stresses in the cement at root canal walls and at the interfaces. The composition of resin cements and different light curing may affect shrinkage stress generated in the root canal. [30]

The aim of this study was to test the influence of the photoactivation timing and the resin cement on the mechanical properties and bond strength of fiber posts to root dentin at different depths. The null hypothesis was that the mechanical properties and bond strength of the resin cements are not influenced by photoactivation timing, resin cement, or their location in the root canal.

METHODS AND MATERIALS

Specimen Preparation

A total of 135 freshly extracted bovine incisors with straight roots and canals were selected for this study and used within one month after extraction. The crowns were removed to obtain a root length of 15 mm. A working length of 14 mm (ie, 1 mm above the apex) was established. The root canals were instrumented by using No. 70 stainless steel K-files in apical thirds (Dentsply Maillefer, Ballaigues, Switzerland) and Gates-Glidden (No. 4 and No. 5; Dentsply Maillefer) in middle and coronal thirds. All instrumentation procedures were followed by 1.0% sodium hypochlorite (NaOCl) irrigation.

Post space preparations were performed in the root canals at a 10-mm depth using conical burs included in the fiberglass post kit (Exacto No. 3; Angelus, Londrina, Brazil). The burs were replaced after every five preparations. The prepared root canals were cleaned thoroughly with tap water and gently dried with absorbent paper points. The fiber posts were luted using one of three dual-cure resin cements: a self-adhesive resin cement, RelyX Unicem (3M ESPE, St Paul, MN, USA) or one of two conventional resin cements, Panavia F 2.0 (Kuraray, Okayama, Japan) with a specific adhesive system for Panavia F, and RelyX ARC (3M ESPE) with the Adper Scotchbond Multi-Purpose adhesive system (3M ESPE) used with the dual-cure mode. The adhesive systems were manipulated and cured following the manufacturers' instructions (Table 1). The posts were etched by immersion in 24% hydrogen peroxide for one minute,[15] rinsed with water for one minute, and dried with an air stream followed by the application of a silane coupling agent for one minute (Silano, Angelus). Resin cements were handled according to the manufacturers' instructions and inserted manually into the root canal with No. 40 stainless steel K-files (Dentsply Maillefer). The fiberglass posts were placed into the root canals with light digital pressure, and the resin cements were photoactivated using one of three different timings (n=15): Immediate (photoactivation immediately after resin cement manipulation, insertion, and excess removal); three minutes (photoactivation delayed for three minutes after the steps described previously); and five minutes (photoactivation delayed for five minutes after the steps described previously). For the three- and five-minute groups the specimens were stored in a dark box. All roots were covered externally with wax to avoid lateral polymerization. Photoactivation was performed through the coronal portion of the root at the buccal, lingual, and coronal faces for 40 seconds, for a total of 120 seconds of light exposure. The photoactivation procedures were performed using a quartz-tungsten-halogen light-curing unit with a 600 mW/cm^2 output (Optilux 501, Demetron Kerr, Orange, CA, USA). The output was periodically measured during the study. After post cementation, the specimens were stored in distilled water at 37°C for 24 hours in a dark plastic container.

Push-out Test

From the 15 specimens prepared for each group, 10 root specimens were randomly selected for "thin-slice" push-out bond strength tests. The roots were sectioned with a precision saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) into six 1.0-mm-thick slabs to obtain two slices each from the coronal, middle, and apical thirds. For the push-out test, the load was applied using a cylindrical tip attached to a mechanical testing machine (DL 2000, EMIC, São José dos Pinhais, Brazil). The diameters of the testing tips (1.5, 1.2, and 0.9 mm) and bases (2.5, 2.2, and 2.0 mm) were selected according to the origin of each slice to accommodate the conical design of the posts and ensure shear stresses along the bonded interface.[32] The load was applied in the apical-coronal direction of the specimens at a crosshead speed of 0.5 mm/min until failure. The bond strength (in MPa) of the post to the root segment was calculated by dividing the load at failure (in N) by the interfacial surface area A of the post section (in mm^2). The lateral surface area of the conical section was calculated using the following formula:

$$A = 2\pi[(R + r)/2]h$$

where $\pi = 3.14$, R is the coronal post radius, r is the apical post radius, and h is the root-slice thickness. The measurements of the root-slice thickness were made using a digital caliper with 0.01-mm accuracy (Mitutoyo, Tokyo, Japan). The radii of the post sections were obtained by capturing an image through a stereomicroscope (Mitutoyo) with a digital camera (Moticam 2300, Motic, Richmond, BC, Canada) and measured with image analysis software (Motic Image Plus 2.0, Motic). The specimens were examined under the stereomicroscope to determine whether the failure mode was adhesive between dentin and post, cohesive in the cement, or mixed.

Vickers Hardness and *E* Determination

Five root specimens from each group were used for determining the *E* and Vickers hardness (VHN) of the cement layer at various levels of the root. The root was sectioned longitudinally into two halves using a precision saw (Isomet 1000, Buehler). One half of each root was randomly selected and embedded in polyester resin (Instrumental Instrumentos de Medição Ltda, São Paulo, Brazil). The surfaces were finished using silicon-carbide papers

Table 1: *Main Composition of the Resin Cements Used in This Study*

Material	Manufacture	Batch Number	Instructions for Use	Composition
RelyX Unicem	3M ESPE, St Paul, USA	421172	1) Dispense cement onto a mixing pad and mix for 20 s; 2) Apply cement in and around canal using endodontic file.	Base: glass powder, methacrylated phosphoric acid esters, TEGDMA, silane-treated silica, sodium persulfate; Catalyst: glass powder, substituted dimethacrylate, silane-treated silica, sodium p-toluenesulfonate, calcium hydroxide
Panavia F 2.0	Kuraray, Okayama, Japan	51213	1) Dispense and mix immediately one drop each of ED PRIMER II; 2) Apply the ED PRIMER into the root canal with a disposable brush tip; 3) After 30 seconds, remove the excess with a vacuum aspirator; 4) Mix sufficient Paste A and Paste B on the paper pad for 20 s; 5) Insert Panavia F manually into the root canal with an endodontic file.	Paste catalyst: Bis-GMA; TEGDMA; glass filler Paste A: silanated silica filler; silanated colloidal silica; MDP; hydrophilic aliphatic D; hydrophobic aliphatic D; dl-camphorquinone; catalysts; initiators Paste B: silanated Ba glass; sodium fluoride; hydrophilic aromatic D; hydrophobic aliphatic D; catalysts; accelerators; pigments (filler content ≅ 76%)
RelyX ARC	3M ESPE, St Paul, USA	N140749	1) Acid etch with 35% phosphoric for 15 s; 2) Rinse with water for 15 s and air dry for 2 s; 3) Remove excess moisture with a paper point; 4) Apply activator of the adhesive system in canal and remove excess with air drying (5 s); 5) Apply primer of the adhesive system in canal and remove excess by air drying (5 s); 6) Apply catalyst of the adhesive system in canal; 7) Dispense cement onto a mixing pad and mix for 10 s; 8) Apply cement in and around canal using endodontic file.	Bis-GMA, TEGDMA, pigments, amine, benzoyl peroxide and zirconia silica (filler content ≅ 67.5%, size < 1.5 um).
Exacto Fiber Post	Angelus, Londrina; Brazil	8214	1) Etch the posts by immersion in 24% H_2O_2 for 1 min; 2) Rinse with water for 1 min; 3) Dry with an air stream; 4) Apply of a silane-coupling agent for 1 min.	Opaque fiberglass post, fiberglass (87%), epoxy resin (13%).

Abbreviations: Ba, barium; Bis-GMA, bisphenol A glycidyl methacrylate; H_2O_2, hydrogen peroxide; MDP, 10-methacryloxydecyl dihydrogen phosphate; TEGDMA, triethylene glycol dimethacrylate.

(600-, 800-, 1200-, and 2000-grit; Norton, Campinas, Brazil) for one minute each and polished with metallographic diamond pastes (6, 3, 1, and 0.25 µm; Arotec, São Paulo, Brazil) for one minute each. The specimens were cleaned using an ultrasound bath with distilled water for 10 minutes after use of each diamond paste. The *E* and VHN of the resin cement layer were assessed with a microhardness indenter (CSM Micro-Hardness Tester, CSM Instruments, Peseux, Switzerland), 24 hours after luting of the fiber posts. The indentations were made at 1.0-mm intervals, starting at 0.5 mm from the coronal surface (coronal third) and ending at 8.5 mm (apical third). The testing procedure was carried out under load control. The load was increased at a constant rate from 0 to 500 mN in 20 seconds (1.5 N/min). The maximum force of 500 mN was applied for five seconds, then the force was gradually decreased to 0

Table 2: *Results for Bond Strength in MPa (n=10)*[a]

Resin Cement	Timing of Light Activation		
	Immediate	3 Min	5 Min
RelyX Unicem	7.6 (3.4) Ac	13.6 (3.9) Ab	18.8 (6.6) Aa
Panavia	6.1 (1.9) Ab	9.5 (3.5) Ba	11.0 (4.0) Ba
RelyX ARC	5.5 (2.5) Ac	9.3 (3.4) Bb	12.2 (4.1) Ba

[a] *Different letters (lowercase for moment of light activation [columns], uppercase for cement [rows]) indicate statistical difference (*$p<0.05$*).*

mN in 20 seconds.[12] The load and the penetration depth of the indenter were continuously measured during the load-unload cycle.

Universal hardness is defined as the test force divided by the apparent area of the indentation at maximal force. From a multiplicity of measurements stored in the manufacturer-supplied database, a conversion factor between universal hardness and VHN was calculated to determine the VHN. The indentation modulus was determined from the tangent of the indentation-depth curve at maximal force. The calculated indentation modulus is comparable with the E of the material.[12]

Post-gel Shrinkage Measurements

Resin cement post-gel linear shrinkage was determined[33,34] using the strain gauge method (n=10). The resin cements (RelyX Unicem, Panavia F 2.0, and RelyX ARC) were manipulated and placed on top of a biaxial strain gauge (CEA-06-032WT-120, Measurements Group, Raleigh, NC, USA) that measured strains during polymerization in two perpendicular directions. The resin cement was polymerized using a quartz-tungsten-halogen unit (Optilux 501, Demetron Kerr) with the light tip placed 1 mm from the surface of the cement. The radiant exposure was set at 24 J/cm^2 (600 mW/cm^2 × 40 seconds). A strain conditioner (ADS0500IP, Lynx Tecnologia Eletrônica, São Paulo, Brazil) converted electrical resistance changes in the strain gauge to voltage changes through a quarter-bridge circuit with an internal reference resistance. Post-gel strains resulting from polymerization shrinkage were monitored for 10 minutes, starting from the beginning of photoactivation. The two perpendicular strain recordings were averaged because the material properties can be assumed to be homogeneous and isotropic on a macro scale. The post-gel shrinkage value at 10 minutes was used in the finite element analysis (FEA).[34] The mean shrinkage strain, which is the linear shrinkage, was reported as percentage volumetric shrinkage by multiplying three times the linear shrinkage by 100%.

Residual Stress Calculation—FEA

To evaluate corresponding residual shrinkage stresses in the root canal, a finite element simulation was carried out using an axisymmetric root restoration model (Figure 1). The geometric model was based on a digitized bucco-palatal cross-section of a maxillary central incisor with cemented fiber post. Coordinates were obtained using ImageJ software (National Institutes of Health, Bethesda, MD, USA). A simplified boundary condition was assumed at the cutting plane of the root (zero-displacement in horizontal and vertical directions). In all models, the root canal was considered as being not root filled (ie, no gutta-percha and sealer) and the posts were considered to be perfectly bonded. The E of dentin[35] was 18.6 GPa and the Poisson ratio was 0.31; the E of the periodontal ligament[36] was 1.18 MPa and the Poisson ratio, 0.45; the E of the bone[35] was 1.37 GPa and the Poisson ratio, 0.30; and the E of the post[37] was 9.5 GPa and the Poisson ratio, 0.34. The E values of the three resin cements using the three photoactivation timings at nine depths were obtained experimentally with the indentation test, as previously described. The Poisson ratio was chosen to be the same[38] for all resin cements at 0.30.

Table 3: *Results of Bond Strength (in MPa) for Each Resin Cement (n=10)*

Root Third	RelyX Unicem				Panavia		
	Immediate	3 Min	5 Min	Pooled Average	Immediate	3 Min	5 Min
Coronal	9.7 (3.8)	16.5 (4.3)	23.6 (6.6)	16.6 (7.6) A[a]	7.8 (1.8) Ab	12.0 (3.1) Aa	14.6 (2.3) Aa
Middle	7.3 (2.7)	13.5 (2.3)	19.3 (5.6)	13.4 (6.1) B	6.0 (1.4) Bb	10.1 (2.9) Ba	11.3 (3.1) Ba
Apical	6.0 (2.7)	10.7 (2.5)	13.5 (3.1)	10.1 (4.1) C	4.6 (1.0) Cb	6.4 (2.0) Cab	7.3 (2.2) Ca
Pooled average	7.6 (3.4) c	13.6 (3.9) b	18.8 (6.6) a		The interaction between factors was significant ($p<0.001$).		

[a] *Different letters (lowercase for activation mode [columns], uppercase for root third [rows]) indicate statistical differences (*$p<0.05$*). In the absence of significant interaction, the comparison is performed for pooled averages.*

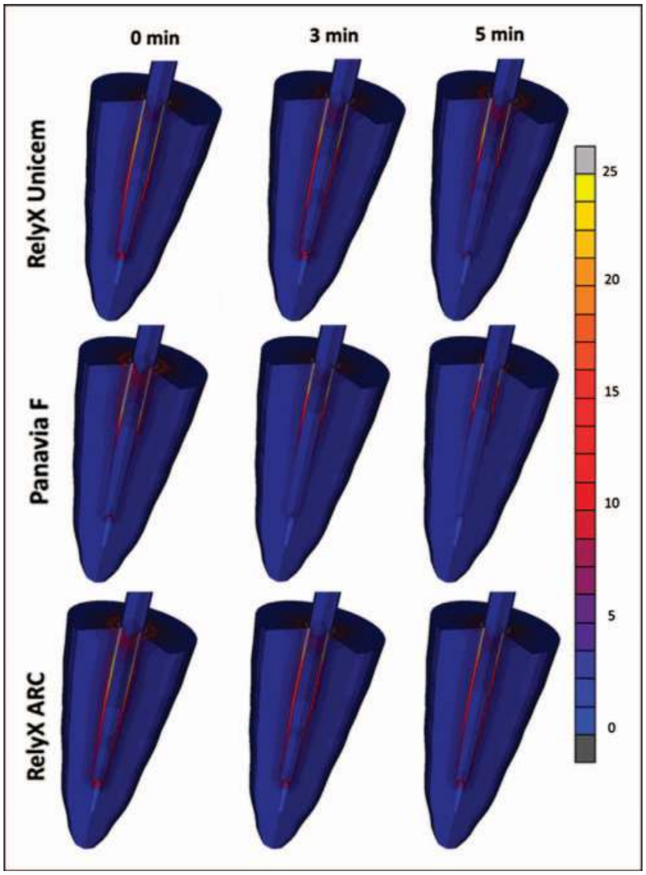

Figure 1. *Residual shrinkage stress distribution (modified von Mises equivalent stress, MPa) for the three resin cements and three photoactivation timings calculated by the numerical polymerization model.*

The FEA was performed using MSC.Mentat (preprocessor and postprocessor) and MSC.Marc (solver) software (MSC Software Corporation, Santa Ana, CA, USA). Nine FEA models were generated with three resin cements (RelyX Unicem [3M ESPE], Panavia F 2.0 [Kuraray], and RelyX ARC [3M ESPE]) and the three photoactivation timings (light curing immediately, three minutes, and five minutes). Polymerization shrinkage was simulated by thermal analogy. Temperature was reduced by 1°C, and the linear shrinkage value (post-gel shrinkage) was entered as the coefficient of thermal expansion. The linear shrinkage values for the coronal, middle, and apical portions of the resin cement were determined by the correlation with the E at each depth.

Modified von Mises equivalent stress, which accounts for the difference in compressive and tensile strengths of the dentin and resin cements, was used to express the residual shrinkage stress conditions in the resin cement nodes at the post and dentin interfaces. Because the finite element program averaged stresses across interfacial nodes, the cement was isolated from the post and the dentin before the nodal values were recorded. Furthermore, stress values were obtained at nodes of the resin cement that corresponded to the nine depths where the E and post-gel shrinkage values were obtained in the experimental test.

Statistical Analysis

Given that push-out bond strength, E, and VHN data were normally distributed (Shapiro-Wilk, $p>0.05$), equality of variance values (Levene test, $p>0.05$) were verified for the push-out bond strength, E, VHN, and post-gel shrinkage before parametric statistical tests were performed. Two-way analysis of variance (ANOVA) in a split-plot arrangement and Tukey test ($\alpha=0.05$) were used to compare push-out bond strength, E, and VHN with the plot represented by the photoactivation timing, resin cement, and their interaction and the subplot represented by the root regions. The post-gel shrinkage values were statistically analyzed by two-way ANOVA followed by Tukey honestly significant difference post-hoc tests ($p=0.05$). The data of failure mode were subjected to the χ^2 test ($p=0.05$). A multiple linear regression analysis was used to determine the influence of the photoactivation timing and root canal third on the bond strength

Table 3: *Results of Bond Strength (in MPa) for Each Resin Cement (n=10) (ext.)*

Root Third	RelyX ARC			
	Immediate	3 Min	5 Min	Pooled Average
Coronal	7.2 (2.6)	12.0 (3.4)	14.8 (3.4)	11.3 (4.4) A
Middle	5.3 (2.2)	9.1 (2.5)	12.5 (3.9)	8.9 (4.1) B
Apical	4.0 (1.6)	6.8 (2.5)	9.2 (3.3)	6.7 (3.2) C
Pooled average	5.5 (2.5) c	9.3 (3.4) b	12.2 (4.1) a	

Table 4: *Percentage of Failure Mode Distribution (%)*

Photoactivation Timing	Resin Cement	Root Region								
		Coronal			Middle			Apical		
		A	M	C	A	M	C	A	M	C
Immediate	RelyX Unicem	60	40	0	65	30	5	75	15	10
	Panavia F	80	20	0	80	15	5	90	0	10
	RelyX ARC	75	25	0	80	20	0	95	0	5
3-min delay	RelyX Unicem	55	45	0	60	40	0	75	15	0
	Panavia F	70	30	0	75	25	0	90	0	5
	RelyX ARC	75	25	0	70	30	0	95	5	0
5-min delay	RelyX Unicem	50	50	0	50	50	0	75	15	10
	Panavia F	70	30	0	75	25	0	90	0	10
	RelyX ARC	60	40	0	75	25	0	95	0	5

Abbreviations: A, adhesive failure between dentin and post; C, cohesive failure in cement; and M, mixed failure.

and also to determine the influence of the photoactivation timing and root canal depth (0.5 to 8.5 in 1.0 mm intervals) in E and VHN data (SAS 9.1 software package, SAS, Cary, NC, USA). The level of significance was set at $\alpha=0.05$.

RESULTS

Push-out Bond Strength

Mean push-out bond strengths are shown in Table 2 and Table 3. A two-factor ANOVA showed that mean push-out strengths were significantly affected by the factors resin cement ($p<0.001$), photoactivation timing ($p<0.001$), and root region ($p<0.001$). There were significant interactions between the factors resin cement and root region ($p<0.001$) and between the factors photoactivation timing and root region ($p=0.002$); however, the interaction between resin cement and photoactivation timing was not statistically significant ($p=0.303$), nor was the interaction among the photoactivation timing, resin cement, and root region ($p=0.700$).

For the immediate photoactivation timing, all resin cements resulted in statistically similar mean push-out bond strengths, irrespective of the root region ($p=0.651$). For the three- and five-minute photoactivation timings, RelyX Unicem showed significantly higher mean push-out bond strengths than other resin cements, irrespective of the root region ($p<0.001$). Comparing the mean push-out bond strengths within each resin cement, RelyX Unicem had significantly higher mean push-out bond strengths for the three- and five-minute photoactivation timings than the immediate curing ($p<0.001$). RelyX ARC and Panavia F cements had significantly higher mean push-out bond strengths for the five-minute timing than for the immediate curing ($p<0.001$). The three- and five-minute photoactivation timings of the Panavia F resin cement had statistically similar mean push-out bond strengths ($p=0.303$).

The multiple linear regression test demonstrated that mean push-out bond strength was significantly associated with the photoactivation timing ($p<0.001$) and root canal depth ($p<0.001$) for all resin cements (Figure 2).

The distribution of failure pattern for each experimental group is shown in Table 4. For all resin cements and photoactivation timings, the adhesive failures were significantly more prevalent at the apical root region and the mixed failures were more prevalent at the coronal root region.

Hardness and E Measurements

Hardness results are shown in Table 5 and Table 6. VHN was significantly affected by the resin cement type ($p<0.001$), photoactivation timing ($p<0.001$), and root regions ($p<0.001$). There were significant interactions between resin cement and root region ($p<0.001$), between photoactivation timing and resin cement type ($p=0.002$), and among resin cement type, photoactivation timing, and root region ($p=0.014$). However, the interaction between photo-

Table 5: *Mean (SD) of Vickers Hardness in N/mm^2*

Resin Cement	Timing of Light Activation		
	Immediate	3 Min	5 Min
RelyX Unicem	40.2 (7.9) ABb[a]	49.2 (2.7) Aa	60.1 (2.8) Aa
Panavia	36.3 (0.8) BCa	36.8 (1.3) Ba	39.1 (2.6) Ba
RelyX ARC	46.1 (1.5) Aa	50.4 (1.3) Aa	51.1 (0.7) Aa

[a] *Different letters (lowercase for moment of light activation [columns], uppercase for cement [rows]) indicate statistical difference ($p<0.05$).*

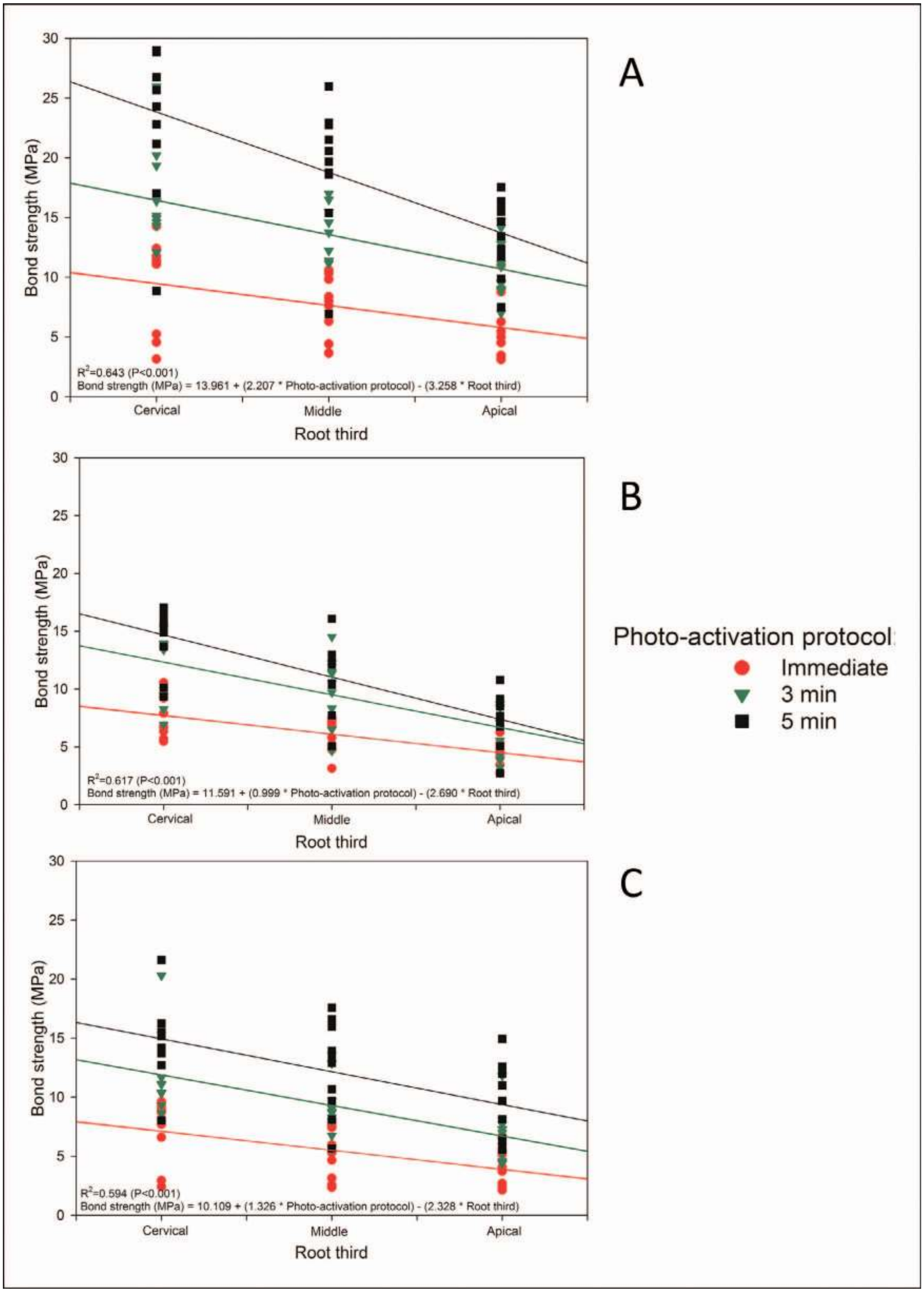

Figure 2. *The relationship between bond strength in root canal and photoactivation timing and root regions. (A): RelyX Unicem, (B): Panavia F, and (C): RelyX ARC.*

Table 6: *Mean (SD) of Vickers Hardness in N/mm^2 for Each Resin Cement (n=5)*

Root Third	RelyX Unicem				Panavia			
	Immediate	3 Min	5 Min	Pooled Average	Immediate	3 Min	5 Min	Pooled Average
Coronal	58.8 (11.2)	66.0 (4.5)	75.5 (4.8)	66.8 (9.7) A[a]	54.8 (3.6)	58.9 (1.3)	64.4 (3.2)	59.4 (5.5) A
Middle	36.0 (6.1)	46.8 (1.9)	62.8 (5.7)	48.5 (12.4) B	35.6 (4.4)	34.8 (2.3)	34.8 (3.3)	35.1 (3.0) B
Apical	25.7 (7.8)	34.6 (2.5)	42.1 (8.9)	34.2 (9.3) C	18.6 (3.3)	16.9 (0.9)	18.2 (1.8)	17.9 (2.1) C
Pooled average	40.2 (16.5) b	49.2 (14.0) b	60.1 (15.7) a		The mode of activation was not significant (p=0.531).			

[a] *Different letters (lowercase for activation mode [columns], uppercase for root third [rows]) indicate statistical differences (*$p<0.05$*). In the absence of significant interaction, the comparison is performed for pooled averages.*

activation timing and root region was not significant ($p=0.738$). In general, the VHN of Panavia F and RelyX ARC were not influenced by the photoactivation timing. RelyX Unicem showed significantly higher mean VHN for the five-minute photoactivation timing than the other two tested timings, irrespective of the root region. Mean VHN decreased gradually from the coronal third to the apical third, irrespective of resin cement.

E results are shown in Table 7 and Table 8. The mean *E* was significantly affected by the resin cement ($p<0.001$), photoactivation timing ($p=0.007$), and root region ($p<0.001$). There is significant interaction between resin cement and root region ($p<0.001$). However, the interactions between resin cement and photoactivation timing ($p<0.3225$), between photoactivation timing and root region ($p<0.700$), and among resin cement, photoactivation timing, and root region ($p=0.310$) were not significant. Mean *E* reduced gradually with root depth for all resin cements. In general, the mean *E* for Panavia F and RelyX ARC was not influenced by the photoactivation timing. However, the mean *E* of RelyX Unicem was significantly higher with the five-minute photoactivation timing.

The multiple linear regression test demonstrated that mean VHN and *E* were significantly associated with the photoactivation timing ($p<0.001$) and root canal depth ($p<0.001$) for all resin cements (Figures 3 and 4). The mean VHN was significantly associated with root canal depth for resin cements RelyX Unicem and Panavia F ($\beta=-5.302$ and -6.702, respectively). Photoactivation timing had significant correlation with VHN values for RelyX Unicem ($\beta=3.913$).

Table 7: *Mean (SD) of Elastic Modulus in Gigapascals (GPa)*

Resin Cement	Moment of Light Activation			Pooled Average
	Immediate	3 Min	5 Min	
RelyX Unicem	5.6 (0.4)	7.4 (0.6)	8.1 (1.1)	7.0 (1.5) A[a]
Panavia	6.5 (0.5)	7.4 (0.3)	7.7 (0.7)	7.2 (0.7) A
RelyX ARC	6.7 (1.2)	6.9 (0.5)	7.3 (0.6)	7.0 (0.8) A
Pooled average	5.8 (1.3) c	6.7 (1.0) b	7.6 (0.7) a	

[a] *For pooled averages, different letters (lowercase for moment of light activation [columns], uppercase for cement [rows]) indicate statistical difference (*$p<0.05$*).*

Mean *E* was significantly influenced by the root canal depth for the resin cement Panavia F ($\beta=1.107$), more than by the depths for RelyX Unicem and RelyX ARC ($\beta=0.644$ and 0.538, respectively). Photoactivation timing had significant influence on *E* for RelyX Unicem ($\beta=0.519$).

Post-gel Shrinkage

The mean values and standard deviations for the post-gel shrinkage of three resin cements and polymerization timings are presented in Table 6. Two-way ANOVA revealed that the volumetric shrinkage mean was significantly affected by the resin cement ($p<0.001$) and photoactivation timing ($p<0.001$). There was also a significant interaction between resin cement type and photoactivation timing ($p=0.001$). For immediate photoactivation, all resin cement showed statistically similar volumetric post-gel shrinkage. The post-gel shrinkage means decreased gradually with delayed photoactivation timing for RelyX and RelyX ARC, but more sharply with Panavia F. As such, the three- and five-minute photoactivation timings for RelyX Unicem and RelyX ARC had significantly higher volumetric post-gel shrinkage mean than those for Panavia F.

Residual Stress Calculation—FEA

Modified von Mises shrinkage stress distributions for all groups are shown in Figure 1. For all cements the immediate photoactivation timing generated higher shrinkage stress than the three- and five-minute timings. Panavia F had the lowest shrinkage stress at the five-minute photoactivation timing of the three resin cements. The delayed photoactivation timing lowered the overall stress levels in the root

Table 6: *Mean (SD) of Vickers Hardness in N/mm^2 for Each Resin Cement (n=5) (ext.)*

Root Third	**RelyX ARC**		
	Immediate	**3 Min**	**5 Min**
Coronal	61.0 (2.7) Aa	59.2 (2.8) Aa	58.5 (0.7) Aa
Middle	44.7 (4.4) Bb	50.1 (2.9) Bab	51.2 (2.5) Ba
Apical	32.6 (2.3) Cb	42.0 (2.1) Ca	43.5 (1.6) Ca
Pooled average	The interaction between factors was significant (p=0.013).		

canal, especially for RelyX Unicem with the five-minute delay.

DISCUSSION

The null hypothesis tested in this study was rejected. The results indicated that the retention of the fiber posts to the root canal dentin measured using the push-out strength was significantly influenced by the photoactivation timing, resin cement type, and root region.

Unlike clinical situations, the root canals in the present study were not filled with sealer and gutta-percha before post space preparation, aiming to eliminate a possible influence of contaminants.[16,39] Gutta-percha and sealers may affect the bond strength of self-adhesive[16] and conventional resin cements.[17,39] In such a case, the influence of the study factors tested would be masked by the presence of sealer and gutta-percha. The impact of the NaOCl and EDTA solutions on bond strength has been controversial in the literature. For conventional and self-adhesive resin cements, the use of NaOCl did not improve the bond strength[17]; however, the use of EDTA solutions can improve the bond strength of conventional resin cements,[17] but not self-adhesive cements.[29] Therefore, the influence of these factors was eliminated in the present study by rinsing the root canals with filtered water only after post preparation.[36,39]

In addition to the bond strength of fiber posts, we also measured the E and VHN of the cements within the root canals, which can provide valuable information indicative of clinical performance.[28] The hardness (VHN) of resin cements is related to degree of conversion when the same material is evaluated and is thus useful for comparing conversion of the resin cements submitted to different photoactivation timings.[40] The E is an important mechanical property that relates stress with functional deformation of materials. E is directly related to the VHN[12] and plays an important role in polymerization stress.[41] Therefore, E and VHN have a direct effect on the bond strength of resin cements used to lute fiber posts to the root canal dentin. E, VHN, and push-out bond strength values of all resin cements, irrespective of the photoactivation timing, decreased from coronal to apical region. The opaque fiber posts used in this study, as well as the translucent posts, do not transmit light along their full length.[12,42] This may have limited the polymerization of the resin cements in the middle and apical root thirds,[20,43,44] resulting in lower push-out bond strength,[7,12,] E, and VHN values in these regions,[12] mainly for Rely X ARC and Panavia F when compared with the self-adhesive resin cement RelyX Unicem.[44] Push-out bond strength and mean VHN measured in the coronal and middle thirds were higher than those measured in the apical third.[40] Thus, the luting of fiber posts with increased lengths should be treated with caution, mainly when the fiber post is cemented with RelyX ARC.[44] The limitation of polymerization in the deeper region of a root canal is reflected in a fracture resistance and structure deformation similar to that of root-treated teeth restored with shorter (5.0 or 7.5 mm) or longer (10.0 mm) posts.[4,46]

The delayed photoactivation timings increased VHN and E values for self-adhesive resin cement (RelyX Unicem) but not for conventional resin cements (Panavia F and RelyX ARC). However, the delayed photoactivation timings increased the push-out strength values for all resin cements evaluated (Figure 2) and reduced the post-gel shrinkage (Table 6). Taking into consideration that VHN and degree of conversion of resin cements are directly related,[40] it can be assumed that the RelyX ARC and Panavia F reached a similar degree of conversion using the immediate and delayed photoactivation timings. The improvement of mechanical properties for RelyX Unicem with delayed light curing may be explained by increased contact of the material with moisture of the root dentin during the curing process.[48] Water plays a critical role in the effectiveness of a self-adhesive resin cement's curing process and bonding characteristics.[48] Additionally, the longer duration

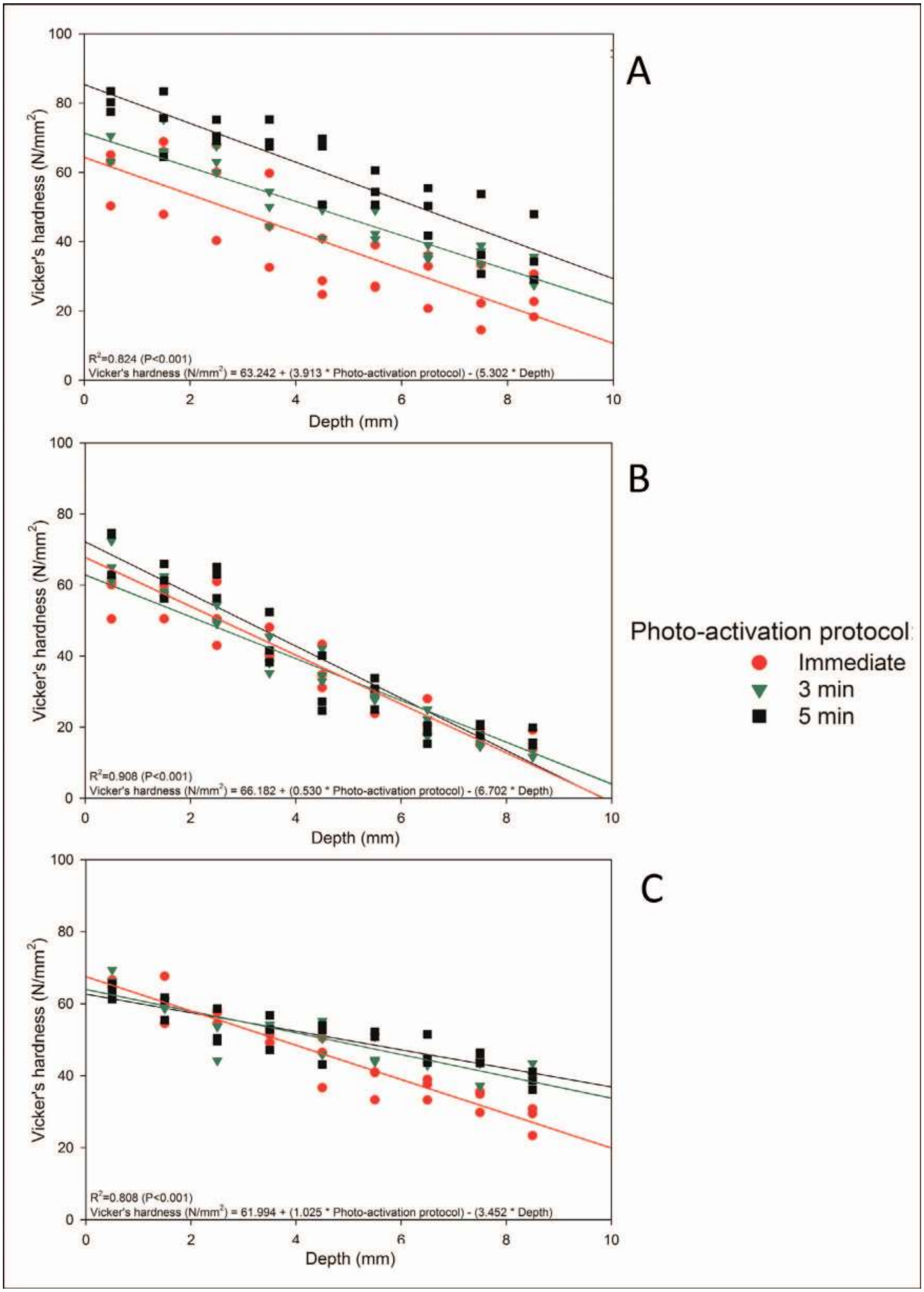

Figure 3. *The relationship between Vickers hardness in root canal and photoactivation timing and root canal depth. (A): RelyX Unicem, (B): Panavia F, and (C): RelyX ARC.*

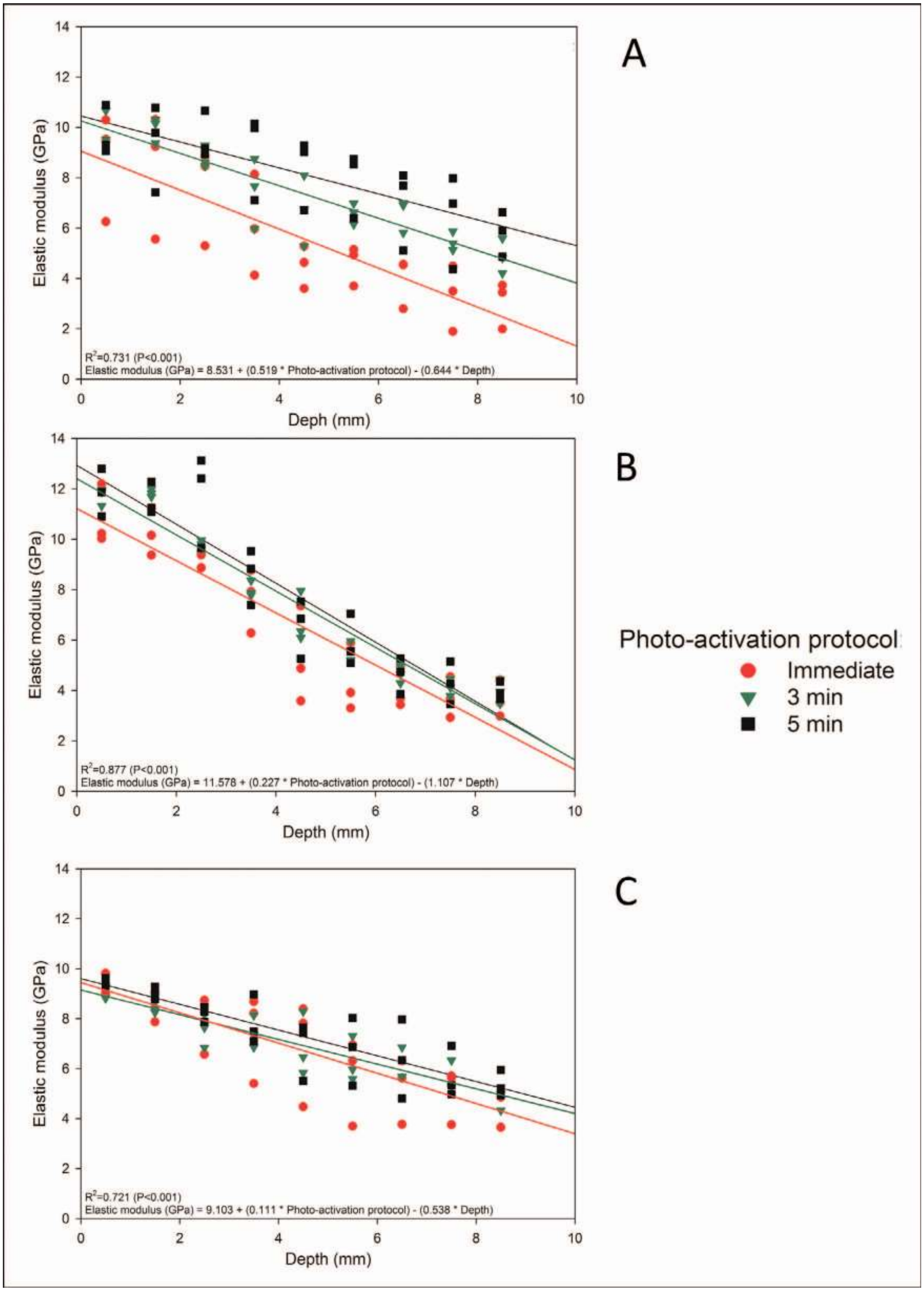

Figure 4. *The relationship between elastic modulus in root canal and photoactivation timing and root canal depth. (A): RelyX Unicem, (B): Panavia F, and (C): RelyX ARC.*

Table 8: *Mean (SD) of Elastic Modulus in Gigapascals (GPa) for Each Resin Cement (n=5)*

Root Third	**RelyX Unicem**				**Panavia**			
	Immediate	**3 Min**	**5 Min**	**Pooled Average**	**Immediate**	**3 Min**	**5 Min**	**Pooled Average**
Coronal	8.2 (2.2)	9.5 (0.5)	9.6 (1.2)	9.1 (1.4) A[a]	10.1 (0.6)	11.1 (0.2)	11.7 (1.1)	11.0 (0.9) A
Middle	5.1 (1.1)	6.9 (1.1)	8.4 (1.5)	7.4 (1.8) B	5.8 (1.3)	6.8 (0.5)	7.0 (0.7)	6.5 (1.0) B
Apical	3.4 (1.1)	5.6 (0.3)	6.4 (1.4)	8.1 (1.8) C	3.8 (0.8)	4.2 (0.2)	4.3 (0.6)	4.1 (0.6) C
Pooled average	The mode of activation was not significant (p=0.108).				The mode of activation was not significant (p=0.218).			

[a] *Different letters (lowercase for activation mode [columns], uppercase for root third [rows]) indicate statistical differences (p<0.05). In the absence of significant interaction, the comparison is performed for pooled averages.*

of chemical activation can be expected to provide a uniform polymerization at the bottom of deep areas where access for curing light is limited. Immediate light activation may limit the interactions of monomers, especially under constrained conditions found in a cavity.[50] Previous studies also showed that delayed photoactivation of RelyX ARC[21,46] and Panavia F[21] did not increase the degree of conversion. This performance can be explained by the presence a chemical activator in the adhesive systems that were used before these resin cements.[49,50] Although the degree of conversion was not affected by the curing timings, delaying photoactivation decreased post-gel shrinkage in this study and polymerization shrinkage stress in a previous study.[46] Lower polymerization shrinkage stress has been related to higher bond strength values.[38] Therefore, delayed photoactivation may not have increased the degree of conversion of the RelyX ARC and Panavia F resin cements, but it could have reduced their polymerization shrinkage stress and consequently increased their push-out strength. Given that most failures of fiber posts are related to debonding,[2,3,5] the higher bond strength found with the five-minute delayed timing is an important finding because it may reduce the incidence of fiber post debonding.

FEA can help to explain the performance of the resin cements cured with the three photoactivation timings because it allows us to study the impact of the resulting different material properties on the stress generation. Given that the FEA models all had the same post/root geometry, the same post/root properties, and the same cement bonding conditions, the stress in the root canal was determined by the combination of post-gel shrinkage and E of the resin cements. In this FEA, the E varied along the post according to location-dependent values that were measured experimentally; whereas, the post-gel shrinkage values for each time were applied along the entire post. The FEA shows that despite the similar or higher E values obtained with delayed photoactivation, residual shrinkage stress levels along the posts decreased. Although not modeled, post-gel shrinkage is likely to decrease the deeper the cement is located,[47] and thus farther down the root canal, the shrinkage stresses can be expected to reduce further. These FEA observations support the previous speculation that delayed photoactivation reduced residual shrinkage stresses and may improve fiber post retention in the coronal region.

The results of *in vitro* studies should be carefully interpreted before being extrapolated to a clinical context. However, the general response of the resin cements to delayed photoactivation timing should also apply under clinical conditions. Therefore, this study presents a potential strategy for clinicians to restore endodontically teeth with delayed photoactivation timing. The current analysis indicates that the best results were found when photoactivation is delayed for five minutes after the manipulation of the resin cements, which is clinically feasible. Shrinkage stress was higher with immediate photoactivation for all resin cements and resulted in higher shrinkage stresses mainly in the coronal third where debonding between resin cement and root dentin account for most failures.

CONCLUSION

Within the limitations of this study, the following conclusions were drawn:

1. The push-out strength of fiber posts to root dentin, E, and VHN of the resin cements were affected by photoactivation timing, resin cement type, and the root canal region.
2. Bond strength increased gradually with delayed photoactivation timing. Significantly higher push-

Table 8: *Mean (SD) of Elastic Modulus in Gigapascals (GPa) for Each Resin Cement (n=5) (ext.)*

Root Third	**RelyX ARC**			
	Immediate	**3 Min**	**5 Min**	**Pooled Average**
Coronal	8.6 (0.7)	8.4 (0.4)	8.9 (0.1)	8.6 (0.4) A
Middle	6.7 (1.8)	6.9 (0.9)	7.2 (1.0)	6.9 (1.2) B
Apical	4.9 (1.1)	5.5 (0.3)	5.8 (0.8)	5.4 (0.8) C
Pooled average	The mode of activation was not significant (p=0.701).			

out strength values were obtained for RelyX Unicem with a five-minute delayed photoactivation timing.

3. The higher push-out strength of the fiber post is achieved on the coronal root region. The bond strengths decreased significantly in the apical region.
4. The residual shrinkage stress decreased with the three- and five-minute delayed photoactivation timings compared with the immediate activation timing.

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Regulatory Statement

The primary institution for this manuscript was the Federal University of Uberlandia.

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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