# Impact of Immediate and Delayed Photo-activation of Self-adhesive Resin Cements on Bonding Efficacy and Water Uptake Under Simulated Pulpal Pressure

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#### Clinical Relevance

A 30-second delay from placement to photo-activation is sufficient to enhance the bonding of self-adhesive cements' functional monomers to dentin while overcoming the deteriorating effect of pulpal fluid flow associated with prolonged delayed photo-activation.

#### **SUMMARY**

This study investigated the effect of immediate versus delayed photo-activation on the bonding

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# performance and water uptake of self-adhesive (SA) resin cements under simulated pulpal pressure (SPP). The occlusal dentin surface was exposed

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in 66 extracted third molars. Resin composite cylinders were cemented to dentin under SPP, with either RelyX Unicem 2 (RU) (3M Oral Care, St Paul, MN, USA) or Maxcem Elite (MC) (Kerr, Orange, CA, USA). Each cement group was equally divided into three groups (n=8 each) according to the time elapsed between placement and photoactivation: immediate activation (IM), 30-second delayed activation (D30), or 120-second delayed activation (D120). Shear bond strength (SBS) was measured, and the type of failure was determined using a stereomicroscope. Three additional samples from each experimental subgroup were used for confocal laser scanning microscopy (CLSM) analysis. A fluorescent dye solution was added to the pulpal fluid reservoir, then a CLSM was used to detect the dye distribution within the tooth-restoration interface. Two-way analysis of variance (ANOVA) and the Tukey post-hoc test were used to analyze the SBS results ( $\alpha$ =0.05). D30 resulted in a significantly higher mean SBS in the two cement groups than IM and D120 (p<0.05). RU showed significantly higher SBS values than MC regardless of the time of light activation (p<0.05). RU showed less dye uptake confined to the cement-dentin interface compared to the MC groups, which showed dye uptake throughout the entire thickness of the cement layer and gap formation at the interface, especially in the D120 group. The 30-second photo-activation delay group significantly improved the bond strength of SA cements. Delaying the photo-activation to 120 seconds increased pulpal fluid uptake by SA cements and compromised the integrity of the bonded interfaces.

#### INTRODUCTION

The clinical performance of contemporary adhesive indirect restorations depends mainly on the successful bonding of adhesive cements to tooth substrates and restorations. <sup>1-4</sup> Nevertheless, given the diversity of their uses in restorative dentistry and the comparatively simple handling technique, many practitioners prefer to use self-adhesive (SA) resin cements over conventional adhesive resin cements. This SA generation does not require any of the steps associated with conventional resin cements, such as acid-etching and the application of adhesive resin. <sup>5,6</sup>

SA resin cements mostly lie in the dual-cured category, combining the advantages of self- and light-cured materials. A significant attenuation of

light occurs while transmitting through large and opaque restorations. Consequently, insufficient light is delivered to the cement. Hence, the polymerization of dual-cure cements depends mainly on their chemical-curing component. Meanwhile, dual-cure cements attain instant and superior polymerization by light-activation at the restoration margins, which is essential for the early retention and stability of the restoration. Moreover, prompt polymerization of the cement at the restoration margins can protect its deeper and internal layers from the oral environment until the slow chemical polymerization is complete. 8,9

Several studies investigated the polymerization kinetics of dual-curing cements and reported conflicting results regarding the impact of time at which the light is applied on the cement properties.<sup>8,10</sup> Although some dual-cured cements attained similar polymerization with and without light-curing, others were negatively affected by insufficient light exposure. 11,12 One study reported that immediate light-curing enhances the degree of polymerization and reduces water sorption of the cement.<sup>13</sup> In contrast, another study concluded that a two-minute delay in photo-activation enhanced the luting agent's conversion.8 Other studies reported that a delay of photo-activation up to five minutes postmixing did not jeopardize the degree of conversion while successfully reducing the polymerization stresses and increasing the bonding efficiency of the cement to the dentin substrate.14,15

The SA cements are hydrophilic in nature and depend on the presence of acid-functionalized monomers to demineralize the tooth surface and chemically bond to the calcium of hydroxyapatite (HAP). Therefore, sufficient time is required for the ionization of these functional monomers. It was reported that immediate photo-activation might turn the resin cement more hydrophobic; hence, delaying the photo-activation may allow a better chemical reaction with the wet dentin substrate. 16,17 Yet, the effect of delayed photo-activation on the bonding performance is material-dependent, due to the difference in the cements' chemical composition, their mechanism of bonding to dentin, and the time needed for the acidic monomers to complete their chemical reaction with the tooth substrate.18 Nevertheless, dentin over-wetness and prolonged fluid movement through the bonded interface may hinder optimal resin seal and can jeopardize the cement's cohesive strength due to excessive water sorption. 19,20

In the current literature, there is a lack of studies considering the effect of simulated pulpal pressure on the bonding efficacy of SA resin cements, when lightcured immediately or after a short delay. Thus, this study was conducted to assess the impact of immediate and delayed photo-activation on the water uptake by two different dual-cured SA resin cements and their shear bond strength (SBS) to dentin under simulated pulpal pressure. The null hypothesis was that photo-activation timing (immediate or delayed) does not affect the bond strength to dentin or the water uptake of dual-cured SA resin cements under simulated pulpal pressure.

#### **METHODS AND MATERIALS**

# Sample Preparation and Simulated Pulpal Pressure (SPP) Setup

Sixty-six extracted human third molars were collected from the oral surgery department at the University of Sharjah Dental Hospital after the university's research ethics committee's approval and patient consent. Teeth that were endodontically treated, carious, prepared, or bleached were excluded from the study. The teeth were cleaned from any soft-tissue remnants using an ultrasonic scaler, then kept in a 1% thymol solution in a refrigerator for one month until used in the study.

The specimens were randomly divided into two equal groups, thirty-three-teeth each, according to the SA resin cement used. The cements used in this study and their composition are summarized in Table 1. Each group was then subdivided according to the time elapsed between the cement and photo-activation application into three equal subgroups.

Each specimen was sectioned transversely using a lowspeed precision diamond saw (Isomet 1000, Buehler, Lake Bluff, IL, USA) under water-cooling to remove the occlusal enamel and expose a flat dentin surface.

The roots of the teeth were then sectioned 1 mm apical to the cementoenamel junction and pulpal tissues were removed using a barbed broach (Dentsply Sirona, Tulsa, OK, USA). The remaining dentin thickness was measured using a pincer-type caliper from the outer dentin surface to the highest pulpal horn, and

it was standardized for all samples to be  $0.8 \pm 0.1$  mm. The pulp chamber was rinsed with a 2.5% sodium hypochlorite solution for 20 seconds and 17% EDTA-solution (pH 8) for 15 seconds, followed by rinsing with distilled water for another 30 seconds to neutralize the effect of the irrigation solutions. Afterwards, the pulp chamber's opening was blocked with red wax and the specimens were embedded in cold-cure acrylic resin in a cube-shaped mold (2 cm edge length). The pulp chamber was exposed by drilling a circular opening (2.5 mm in diameter) on the mold's bottom side, and the wax was removed using a hot instrument. Specimens were then kept in distilled water at room temperature to prevent dehydration.

Under water-cooled, 600-grit SiC abrasive papers were used in a polisher/grinder (EcoMet 30, Buehler) to create a uniform smear layer on the dentin surfaces. The specimens were then rinsed with double-distilled water, cleaned in an ultrasonic water bath, and kept hydrated in containers filled with double-distilled water. A Tygon tube with a 2.5-mm external diameter and 1.5-mm internal diameter (Sigma-Aldrich, Buchs, Switzerland) was inserted in the pulp chamber opening and secured with cyanoacrylate super glue (Scotch Super Glue Liquid, 3M, Maplewood, MN, USA) to seal any gap between the tube and the specimen surface. Care was taken to avoid filling the pulp chamber with glue. A 20-mL syringe was filled with deionized water and connected to the Tygon tube and was suspended 20 cm above the sample's level to simulate 20-cm H<sub>2</sub>O pulpal pressure.

### Cementation and Shear Bond Strength (SBS) Test

Eight samples from each sub-group were used for the SBS test. Pre-polymerized resin nano-composite cylinders (Filtek Z350 XT Universal Restorative, 3M Oral Care, St Paul, MN, USA) were prepared using a Teflon mold (2.0 mm in diameter and 2.0 mm in height).

Table 1: Self-adhesive Resin Cements Evaluated: Their Composition and Respective Manufacturer's Information						
Cement	Abbreviation	Manufacturer	er Composition			
RelyX Unicem 2 Automix	RU	3M Oral Care, St Paul, MN, USA	Base paste: phosphoric acid-modified methacrylate monomers, bifunctional methacrylate acetate, initiators, stabilizers, glass fillers, silica, calcium hydroxide	430451		
			Catalyst paste: methacrylate monomers			
Maxcem Elite	MC	Kerr Dental Orange, CA, USA	GPDM, methacrylate ester monomers, HEMA, 4-methoxyphenol, cumene hydroperoxide, titanium dioxide and pigments	3441545		
Abbreviations: GPD, glycerol phosphate dimethacrylate; HEMA, 2-hydroxyethylmethacrylate.						

For all groups, the dentin surface was gently dried with a stream of air. Then the cylinders were cemented to the dentin surface using either RelyX Unicem 2 Automix (RU, 3M Oral Care) or Maxcem Elite (MC, Kerr Dental, Orange, CA, USA). For both cements, the cement base and catalyst were mixed using an automixing tip and applied using an ultrafine 1-mm tip. The cements were applied on the dentin surfaces and the composite cylinders were placed over the cement layer at the center of the dentin surfaces. During the cementation of each resin composite cylinder, a special metal clip was used to stabilize the cylinder and induce a standardized load for 20 seconds. The excess cement was removed around the margins using a microbrush. From the beginning of the mixing until the complete removal of the excess cement, the total placement time was set to 40 seconds per specimen. The metal clip was removed carefully, and each sub-group (n=8) received one of the three following light-curing protocols: 1) Immediate (IM) – the cement was immediately lightcured after placement; 2) Delayed 30 seconds (D30) the cement was light-cured 30 seconds after placement; or 3) Delayed 120 seconds (D120) - the cement was light-cured 120 seconds after placement.

Photo-activation was done from the top surface of the resin cylinders for 20 seconds using an LED lightcuring unit (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) operating at 1200 mW/cm<sup>2</sup> in standard mode. The intensity was verified every eight samples using a digital radiometer (Cure Rite, Dentsply, Milford, DE, USA) to ensure uniform curing. After the cementation procedures, all specimens were stored in distilled water at 37°C for 24 hours to allow for cement maturation. Then, the SBS was tested using a table-top shear bond tester (Bisco Inc, Schaumburg, IL). Shear forces were applied on the tooth-restoration interface by a semicircular metal attachment that ran at 1 mm/min until complete failure of the cement and debonding. The force at the time of failure was recorded, and the bond strength was calculated by dividing the force at failure by the surface area of the bonded interface to get SBS in MPa using the following equation:

SBS (MPa) = Force (N) 
$$/$$
Area (mm<sup>2</sup>)

The specimens were then examined under a stereomicroscope (Model SM-3BX-80S, AmScope, Irvine, CA, USA) with magnification from 3.5X – 4.5X to determine the type of failure: whether cohesive failure within the cement (type I), an adhesive failure between the cement and the tooth (type II), or a mixed failure (type III).

# Confocal Laser Scanning Microscopy (CLSM) Analysis

Three additional samples from each experimental sub-group were used for CLSM analysis. A 0.1 wt% fluorescein sodium salt dye solution in deionized water (Sigma-Aldrich, St. Louis, MO, USA) was added to the simulated pulpal fluid reservoir. After preparation and cementation procedures under SPP, the remaining dye in the pulp chamber was thoroughly washed out with water. The top surfaces of the specimens were embedded in a cold-cure acrylic resin. Afterwards, the specimens were stored in distilled water for 24 hours. After storage, the specimens were sectioned longitudinally in a direction perpendicular to the bonded interface with a slow-speed diamond saw running under water-cooling (Isomet 1000, Buehler) to obtain a 1-mm-thick section from the center of the tooth. The longitudinal sections were polished using 1200-grit SiC abrasive papers and placed in an ultrasonic water bath for five minutes to remove loose debris. The sections were mounted on glass slabs, then the tooth-restoration interfaces were examined immediately with a confocal laser scanning microscope (Nikon Eclipse Ti-S, Nikon Instruments Inc, Melville, NY, USA). Microscopic images were taken for the tooth-restoration interface using 468 nm laser illumination, with up to 20x magnification. The fluorescent dye distribution within the interface and the dye uptake by the cement layer were assessed.

#### **Statistical Analysis**

The data obtained from the SBS testing were analyzed by SPSS software (IBM SPSS statistics V24.0, IBM Corp, Armonk, NY, USA). Two-way analysis of variance (ANOVA) was used to assess the effect of different photo-activation timings and cement type on the SBS of the cements tested. Tukey post-hoc test was used to detect pairwise differences among the experimental groups. A 95% confidence level was applied for all the statistical tests ( $\alpha$ =0.05), and the power of the study was 0.90. The Chi-square test was used to compare between failure modes of the tested groups.

#### **RESULTS**

#### **Shear Bond Strength (SBS)**

Mean SBS values and standard deviations (SD) of the experimental groups are presented in Table 2. Two-way ANOVA revealed a significant effect of both photoactivation timing and the cement type on SBS to dentin (p<0.001). The results showed that, in the two tested resin cement groups, D30 resulted in a statistically significant higher mean SBS compared to IM and D120

Table 2: Mean Shear Bond Strength Values (MPa ± Standard Deviation) of RelyX Unicem 2 Automix (RU) and Maxcem Elite (MC) After Different Dwell Times<sup>a</sup>

Dwell	Cement Type				
Time	RU	MC			
IM	12.3 ± 3.2 Aa	3.9 ± 1.1 Ba			
D30	22.4 ± 6.4 Ab	7.6 ± 2.6 Bb			
D120	13.6 ± 6.2 Aa	4.8 ± 1.6 Ba			

Abbreviations: D30, delayed 30 seconds; D120, delayed 120 seconds; IM, immediate; MC, Maxim Elite; RU, RelyX Unicem 2 Automix.

(p<0.05). Moreover, RU showed significantly higher SBS mean values compared to MC (p<0.05), regardless of the photo-activation timing applied.

Failure modes of the SBS tested specimens are summarized in Table 3. The Chi-square test showed that there was a statistically significant difference between failure modes of the two materials (p<0.001). Under the three photo-activation timings, the predominating type of failure was cohesive in RU groups and adhesive in MC groups.

#### Confocal Laser Scanning Microscopy (CLSM)

Representative images of the CLSM analysis are presented in Figure 1. Fluorescent dye uptake was noted in all tested groups. Still, its extent and manifestation varied according to the cement type and the time elapsed before photo-activation. In the IM specimens of RU, the dye uptake was confined to the cement-dentin interface and within the thin hybrid layer (Figure 1A), while the hybrid layer was thicker in the D30 group, up to 10  $\mu$ m from the adhesive interface (Figure 1B). In addition, no gap formation was noticed in these two groups. In contrast, in D120, the dye uptake was more obvious, showing a structureless pattern that varied in width but was approximately 50  $\mu$ m at its widest, and discrete gaps were found at the dentin-cement interface (Figure 1C).

In the MC groups, the dye uptake increased with extending the waiting time before photo-activation, and it was generally more obvious than that of RU groups. The dye uptake in the IM group was more scattered and less obvious compared to the more noticeable and uniform distribution of the dye along the adhesive interface in D30 and D120 groups (Figure

Table 3: Failure Type Percentage of RelyX Unicem 2 Automix (RU) and Maxcem Elite (MC) After Different Dwell Times

	Failure	Dwell Time		
Material	Failure Type	IM N (%)	D30 N (%)	D120 N (%)
RU	Cohesive	5 (62.5)	6 (75.0)	6 (75.0)
	Adhesive	0 (0)	1 (12.5)	0 (0.0)
	Mixed	3 (37.5)	1 (12.5)	2 (25.0)
MC	Cohesive	3 (37.5)	1 (12.5)	0 (0.0)
	Adhesive	3 (37.5)	6 (75.0)	6 (37.5)
	Mixed	2 (25.0)	1 (12.5)	2 (25.0)

Abbreviations: D30, delayed 30 seconds; D120, delayed 120 seconds; IM, immediate; MC, Maxim Elite; RU, RelyX Unicem 2 Automix

1D). Moreover, D30 showed a scattered dye infiltration through the cement layer that reached the cement-restoration interface (Figure 1E). D120 showed a highly stained thick zone invading the entire thickness of the cement layer, with complete separation at the adhesive interface (Figure 1F).

#### **DISCUSSION**

This study was conducted to evaluate the effect of the time elapsed between the placement of two dual-cured SA resin cements and light-activation on their bonding efficacy to both the dentin substrate and the restoration by assessing the SBS in the presence of SPP followed by determining the failure mode. In addition, CLSM was utilized to examine the dye uptake extension and pattern at the tooth-restoration interface. The results of this study revealed a significant effect of the photoactivation timing on the SBS of the tested cements; thus, the null hypothesis was rejected.

In the current study, the tested cements were either photo-activated immediately, after 30 seconds or after 120 seconds following their placement. The authors chose these delay periods as they considered them more clinically acceptable than those proposed by previous studies, in which the delay in photo-activation was up to 10 minutes.<sup>8,21</sup>

There is a great divergence of views in the literature regarding the effect of time elapsed between applying the dual-cured cements on dentin and their photoactivation. While delaying the photo-activation may not increase the polymerization potential of resin-based cement, its ability to reduce the induced polymerization stress and increase the bond strength to dentin has been shown in some studies. <sup>21,22</sup> A study by Fonseca

<sup>&</sup>lt;sup>a</sup>Within a row, same uppercase letters show mean values with no statistically significant difference (p>0.05). Within a column, same lower-case superscript letters show mean values with no statistically significant difference (p>0.05).

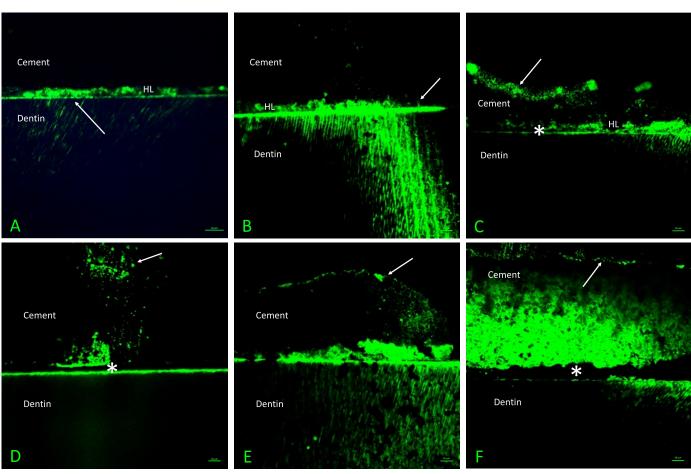


Figure 1. Representative confocal laser scanning microscopy (CLSM) fluorescence images showing the green, fluorescent dye uptake under simulated pulpal pressure. A) RelyX Unicem 2 Automix (RU) immediate photo-activation; dye uptake within the thin hybrid layer (HL) and the dentin adhesive interface (white arrow); B) RU with 30-sec delay; dye uptake within the wider HL and the dentin adhesive interface (white arrow); C) RU 120-sec delay; a deeper dye uptake, up to 50 µm within the cement layer (white arrow) and discrete gaps at the adhesive interface (asterisk); D) Maxcem Elite (MC) Immediate photo-activation; separation at the adhesive interface (asterisk) and discrete dye uptake through the cement layer (white arrow); E) MC 30-sec delay; uniform dye distribution along the HL and reaching the top surface of the cement layer (white arrow); and F) MC 120-sec delay, severe dye uptake through the entire thickness of the cement layer and reaching to the top surface (white arrow), complete separation at the dentin-cement interface (asterisk).

and others investigated the effect of delayed photoactivation on SA resin cement's degree of conversion and water sorption; the authors concluded that the photo-activation timing modified the behavior of the cement and that the dual-cured SA resin cements should be photo-activated as soon as possible after placement.<sup>13</sup> Unfortunately, in most of these studies, testing the properties of SA cements was not done under SPP; hence, the effect of dentinal fluids on the bonding efficacy of the cement was not considered.

Within the two investigated cement groups, the results of our study showed that the mean SBS was significantly higher for the D30 groups than the IM and D120 groups. Self-adhesive cements bond to the tooth tissues through a twofold mechanism: micromechanical interlocking and chemical bonding. The first is created by the acidic monomers that partially demineralize the dentin and

simultaneously infiltrate the collagen network, creating a hybrid layer. While the second mechanism is slowly established through chemical bonding between the self-adhesive functional monomers and the remaining HAP around the demineralized collagen within the relatively thin hybrid layer.<sup>23,24</sup> According to the "adhesion-decalcification" (AD) concept that was described by Yoshida in 2004, functional monomers like 10- methacryloyloxydecyl dihydrogen phosphate (10-MDP) bond electrostatically to HAP, creating a primary chemical bond to the calcium of HAP and forming a characteristic self-assembled nano-layered structure.25 In our study, RelyX Unicem was chosen as a representative of the MDP-based SA cements, because it is the most thoroughly investigated SA cement in the literature. Similarly, most of the previous studies done on non-MDP-based SA cement used Maxcem Elite.<sup>26</sup>

We postulate that a 30 second-delay in light-curing allowed the functional phosphoric acid esters, 10-MDP in RU, and glycerol dimethacrylate dihydrogen phosphate (GPDM) in MC, to sufficiently demineralize the dentin surface, optimizing the micro-mechanical adhesion. In addition, this 30-second delay of photo-activation might have permitted the functional monomers to interact with the calcium of HAP and eventually resulted in a significant nano-layering without being interrupted by photo-activation.

GPDM was utilized for a long time in etch-and-rinse adhesives, such as Optibond FL (Kerr), and exhibited high bond strength to dentin, both immediately and upon aging.<sup>27,28</sup> Subsequently, GPDM was added as a functional monomer to self-etch adhesives, SA cements, and SA restorations. The bonding performance of GPDM-based SA restorations was reported to be inferior to that of MDP, due to its high hydrophilicity. 28,29 these findings are consistent with our results, which revealed that RU showed significantly higher (\$\phi<0.05) SBS mean values compared to MC regardless of the photo-activation timing applied. The superior performance of RU can be attributed to its ability to form nano-layering of stable 10-MDP-Ca salts and a durable bond to HAP, compared to the reported unstable bond of GPDM to dentin.27-31 Further explanation was described by Van Meerbeek and others, who described a submicron HAP-rich hybrid layer formation without obvious collagen exposure by the 10-MDP-based adhesives. 32,33 In contrast, a parallel study by Yoshihara and others reported that GPDMbased adhesives yielded a thicker (1.5 to 2 µm) HAPpoor hybrid layer with visible collagen exposure.<sup>28</sup>

Light-activation of dual-cured cements induces fast polymerization, which produces a large number of free radicals. The early vitrification by the light-activation causes the entrapment of the free radicals within the organic matrix, and hence minimizes the extent of the subsequent self-polymerization of these cements, which compromises the overall degree of conversion of the polymer.<sup>8,34</sup> This may explain the low bond strength values obtained from the immediately light-cured groups. This justification may suggest that delaying the light activation procedure would result in a higher degree of conversion of the cement, and consequently, a higher bond strength. This postulation may be true until a certain point is reached, when the chemical curing time becomes a limiting factor due to the cement reaching a high level of viscosity (vitrification point), restricting the mobility of the unreacted monomers.<sup>11</sup>

In the current study, both cements exhibited lower mean SBS values with a photo-activation delay of 120 seconds compared to those reached by a 30-second delay. These results might further emphasize the impact of reaching and/or exceeding the aforementioned limiting point in the time spectrum of complete polymerization on the bond strength. Moreover, although the acidic functional monomers are mostly consumed by the reaction with the HAP of the tooth, the remaining unreacted acidic monomers are believed to inhibit chemical polymerization by their deactivating effect on free radicals. Therefore, if insufficient polymerization is achieved by photo-initiation at a certain point, the self-curing process may be significantly compromised.<sup>5</sup>

It is generally described that a serum-like fluid fills the dentinal tubules and flows from the pulp chamber by hydrodynamic pressure of approximately 24 cm Hg in vital teeth. The egression of pulpal fluid has been shown to compromise the bonding of adhesive systems.<sup>35</sup> The effect of simulated pulpal pressure on the bonding characteristics of SA cements was confirmed by our CLSM findings, as the fluorescent dye intake generally increased with delaying the photo-activation timing. The immediate curing groups showed limited dye uptake, which was only confined to the restoration/ dentin interface area (Figure 1A and 1D). In contrast, the results may indicate that if the egress of the pulpal fluids under pressure is maintained for a relatively long time, it may cause separation between the cement and the dentin substrate. Additionally, the pulpal fluid can infiltrate through the entire thickness of the cement layer and may reach the cement-restoration interface, as shown at the adhesive interface of the D120 groups of MC (Figure 1F).

The prolonged contact between the uncured hydrophilic monomers and the dentin surface may increase the dentinal fluid uptake within the adhesive interface, negatively influencing the cement polymerization and bonding efficacy.<sup>36</sup> This effect might explain the lower bond strength values and a higher percentage of cohesive failures within the D120 groups.

Our study's findings are in agreement with those reported by Hiraishi and others, who concluded that the pulpal fluid permeation during the initial setting period could deteriorate the bonding quality of resin cement. This deteriorating effect was signified as reduced microtensile bond strengths and porous bonding interfaces through the cured adhesive layer. In contrast, Mazzitelli and others indicated that the SPP effect is material-dependent; while some SA cements, namely RU and Bis-Cem (Bisco), had a significant increase in their bond strength under SPP, other cements were not significantly influenced by the presence or absence of SPP. The authors explained that the lack of water in the RU composition justified

the essential role of the water from the pulpal fluid and wet dentin in ionization of the phosphoric acid esters, which enable the phosphoric acid to react with the alkaline fillers and tooth apatite, leading to better setting and improved bonding.<sup>16</sup>

The predominate type of failure in RU groups was cohesive in nature. The authors cannot attribute these results to the materials' immoderate water uptake, as the CLSM analysis revealed lower water uptake by RU compared to MC. Alternatively, these results can be credited to the established strong adhesive bond with the dentin substrate, which may have surpassed the cohesive strength of the cement. Similar results were reported by Mazzitelli and others, who showed a higher percentage of cohesive failures within the SA cements' layers under SPP than when no SPP was applied. Another study showed that under SPP, the predominant type of failure noticed in RU was cohesive, while the highest percentage of failure in all other investigated cements was adhesive. 17

One limitation of the current study is that it assessed the immediate bond strength of the tested materials (after 24 hours storage). Although the results of our study may be predictive of the long-term performance of the tested SA cement under deteriorating oral environmental conditions, aging the samples under wet storage conditions, alone or with thermal and/or mechanical loading, could have given more clinically relevant information.

#### CONCLUSION

Under SPP testing conditions, it can be concluded that a 30-second delay after the placement of the SA cement on dentin is recommended before photo-activation. This time is sufficient for the SA functional monomers to complete their chemical reaction with dentin. Simultaneously, this delay period is short enough to avoid the pulpal fluid's deteriorating effect associated with the prolonged delay of photo-activation. Compared to GPDM-based cements, MDP-based cement showed superior bond strength and less pulpal fluid uptake, regardless of the photo-activation delay time applied.

#### **Regulatory Statement**

This study was conducted in accordance with all provisions of the human subjects oversight committee guidelines and policies of the University of Sharjah Research Ethics Committee (approval code REC-18-10-09-03-S).

#### **Conflict of Interest**

The authors of this article 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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