Does curing delay affect the bond strength of fiber post with self-adhesive cements?

Allegra Comba, DDS PhD, Andrea Baldi, DDS, Elvinas Juzikis, Edoardo Alberto Vergano, DDS, Damiano Pasqualini, DDS, Mario Alovisi, DDS PhD, Elio Berutti, MD DDS, Nicola Scotti, DDS PhD

a PhD, Department of Biomedical and Neuromotor Sciences, University of Bologna, Bologna, Italy.
b PhD student, Department of Surgical Sciences, University of Turin, Turin, Italy.
c Post graduate student in Prosthodontic, Lithuanian University of Health Sciences, Medical Academy, Faculty of Odontology
d Clinical Tutor, Department of Surgical Sciences, University of Turin, Turin, Italy.
e Associate Professor, Department of Surgical Sciences, University of Turin, Turin, Italy.
f Assistant Professor, PhD, Department of Surgical Sciences, University of Turin, Turin, Italy.
g Dean, Full Professor, Department of Surgical Sciences, University of Turin, Turin, Italy
h Associate Professor, PhD, Department of Surgical Sciences, University of Turin, Turin, Italy.

Corresponding Author:
Prof. Nicola Scotti,
Via Nizza 230, 10126, Turin, Italy
Tel: +39-340-2861799
Fax: +39-011-6620602
e-mail: nicola.scotti@unito.it

Declaration of interest: none.
ABSTRACT

**Purpose:** To evaluate the effect of three different curing protocols based on different ratios between self-curing and light-curing periods on the bond strength and nanoleakage of fiber posts luted with dual-curing self-adhesive cements. **Materials and Methods:** A total of 48 single-root teeth were endodontically treated and obturated, and an 8-mm post space was prepared with dedicated drills. Specimens were randomly divided into two groups according to the self-adhesive cement employed: G1 = PANAVIA SA Plus (Kuraray Noritake); and G2 = Bifix SE (VOCO). The specimens were further divided into three subgroups (n = 8 each) according to the light curing protocol applied: no light curing (SG1); 20 seconds of light-curing 20 seconds after cement injection (SG2); and 20 seconds of light-curing 120 seconds after cement injection (SG3). Slices of 1-mm thickness were prepared to perform the push-out test and nanoleakage analyses of the coronal and apical regions after 24 hours of storage in artificial saliva. Results were statistically analyzed with three-way ANOVA and Tukey post hoc tests. Statistical significance was set for $P < .05$. **Results:** Three-way ANOVA analysis showed that the factors cement ($P = .02$) and curing protocol ($P < .001$) had a significant influence on bond strength. Tukey post hoc test reported that light curing 120 seconds after injection showed higher bond strength compared to no light curing and photoactivation after 20 seconds. **Conclusion:** To achieve the highest bond strength with self-adhesive cements, photoactivation with a 120-second delay after mixing is required. Nevertheless, there is no difference if the time elapsed before applying the light is 20 seconds or if there is no light curing at all. *Int J Prosthodont 2021. doi: 10.11607/ijp.7486*
INTRODUCTION

Endodontically treated teeth (ETT) are usually associated with extensive loss of dental crown structure, which might require the use of radicular post and core to retain the final restoration. In recent years, glass-fiber posts (GFPs) applied through adhesive procedures have significantly increased their popularity at the expense of metal posts, mainly thanks to their elastic modulus, which provides favorable stresses distribution within the root.\(^1\)

A recent clinical study reported higher long-term survival rates for endodontically treated teeth that were restored with GFPs (94.3%) when compared to teeth that did not receive GFPs (76.3%).\(^2\) Nowadays, fiber post placement requires adhesive procedures that are technique-sensitive, with several anatomical and clinical factors which can potentially affect the adhesion to root dentin such as histological and anatomical characteristics of the root canal, orientation and density of dentinal tubules in different root canal area,\(^3\) and operator accessibility to the post space.\(^4-7\) Some of these factors could not be easily limited and attenuated by the control of clinical steps and are strongly related to the root canal itself.\(^7\) Among them, a great impact is due to the cavity configuration factor (C-factor) of an endodontic post space, which makes adhesion highly unfavorable due to the small amount of unbonded surface areas in the root canal that cause insufficient stress relief for luting cements during polymerization. During the pre-gel phase, shrinkage stress is very low due to the high flowability of the resin material, but when the gel phase is reached, its flowability is lost and the stress generated by volume shrinkage is transferred to the root canal walls, possibly causing immediate adhesive or cohesive failures. Several authors demonstrated that in radicular dentin shrinkage stresses might exceed bond strength, resulting in gaps at the interface, loss of retention, and displacement of the GFP.\(^6,8\)
Considering the difficulties in obtaining a correct and durable adhesion to radicular dentin, the most common failures of GFP-supported restorations are represented by debonding or leakage\textsuperscript{9,10} occurred during the luting process\textsuperscript{11}.

Recently, in order to simplify, standardize and minimize the side effects of the clinical scenario of GFPs luting procedure, self-adhesive resin cements were introduced in the dental market to reduce technique-sensitive steps such as acid-etching, priming, and bonding\textsuperscript{12}. These materials possess acid-functionalized (meth)acrylate monomers with carboxylic or phosphoric acid-groups, which have the propriety of demineralizing enamel and dentin and create a hybrid layer due to their initial pH. The calcium obtained from the hydroxyapatite is then bonded by the same ionized functional acidic-(meth)acrylates to the resin network\textsuperscript{13-15}. In this way, chemical bonding is achieved without the risks of over-etching, over-drying, or over-wetting of dentin collagen fibers\textsuperscript{16}.

The process to achieve interaction with dentin is not instant and it has been suggested that self-etch adhesive systems may require at least 20s scrubbing before curing procedure to improve resin monomer infiltration into dentinal substrate. Indeed, the formation of a solid and stable hybrid layer is obtained thanks to the chemical reaction that occurs when the acidic monomer interacts with the smear layer and produces a pH variation of the system\textsuperscript{17}.

However, little information is available regarding how the curing kinetic and protocols of self-adhesive cements and the related pH neutralization could affect the GFP bond strength. Some authors claimed that light activation could negatively affect self-curing mechanism\textsuperscript{8,18}. The rationale is that the rapid formation of a cross-linked polymer after light exposure would lead to entrapment of the reactive species, including activators and initiators needed for self-curing reaction\textsuperscript{18}.
Thus, the aim of the present in vitro study was to evaluate the effect of different curing protocols based on three different waiting time before light-curing activation, on radicular bond strength and nanoleakage of fiber posts luted with dual-cured self-adhesive cements. The null hypotheses tested were that the bond strength and the nanoleakage are not influenced by related to the self-adhesive cements (1), the curing protocol (2), and the post space region (3).

METHODS AND MATERIALS

Study design

A general description of the main materials used in the present study, their manufacturers, and composition are listed in Table 1.

Push-out bond strength was designed with two main groups (n=24 each) according to the self-adhesive cement material and three subgroups (n=8 each) according to the polymerization protocol applied. The nanoleakage was designed with eighteen (n=18) samples, meaning three for each subgroup (n=3 each) as previously described.

Specimens preparation

Forty-eight (n=48) human single-rooted teeth extracted for periodontal reasons were selected for the present study. The inclusion criteria were the absence of radicular caries or fractures, no cracks under transillumination and 20x optical microscope magnification, no previous endodontic, restorative, or prosthetic treatment involving the root, and similar and linear root anatomy with a minimum root length of 13±3 mm. Specimens were stored in 1% chloramine-T solution at 4 °C and used for the test within one month after extraction. Anatomical crowns were removed using a low-speed diamond saw under water-cooling (Micromet, Remet). Teeth without a round circular-shaped canal system were excluded from the present study.
Endodontic treatment was carried out in all specimens by the same expert operator using Waveone® GOLD (Dentsply Maillefer) to the working length, set at 1mm short of the visible apical foramen. Irrigation was performed with 5% NaOCl (Niclor 5, Ogna) alternated with 17% EDTA (Tubuliclean, Ogna). After water rinsing and drying with paper points, canals were obturated with gutta-percha points (GuttaPercha Points Medium, Inline) using Down Pack heat source (Hu-Friedy, Chicago, IL, USA) and an endodontic sealer (Pulp Canal Sealer EWT, Kerr). Backfilling was performed with the Obtura Max system (Analytic Technologies). Specimens were then stored in distilled water for seven days and randomly allocated for group division (https://www.randomizer.org/).

A standardized 8mm length post space was prepared in each specimen using dedicated burs #2 (Voco GmbH). The adaptation and length of the selected glass fiber posts (Rebilda Post #15, Voco GmbH) were verified and approved by the same expert operator. Each fiber post was cleaned for 30s with ethanol. Specimens were then randomly divided into two (n=24) according to the self-adhesive cement employed: Panavia SA Cement Plus (Kuraray Noritake) for Group (G1) and Bifix SE (Voco GmbH) for Group 2 (G2).

For both groups, manufacturer instructions were followed: Once post-space was perfectly dried through air syringe and paper cones, self-adhesive cement was automatically mixed in the dedicated syringe and inserted into the root canals from apical to coronal portion. A little amount of cement was also placed in the apex of the fiber posts, and then the posts were gently inserted into the post-space, with a slight turning motion in order to prevent air bubbles according to the instructions. Specimens were then randomly divided (https://www.randomizer.org/) into three subgroups (n=8) according to the light-curing protocol applied: no light-curing (Subgroup 1, SG1); 20s light-curing after 20s from cement injection (Subgroup 2, SG2); 20s light-curing after
120s from cement injection (Subgroup 3, SG3). A poly-wave LED curing light (Translux Two-Waves, Kulzer) at 1400 mW/s was employed for all samples. Specimens were then stored in distilled water for 7 days.

**Push-out bond strength test**

All specimens underwent push-out bond strength evaluation. Specimens were cut into transverse sections using a low-speed diamond saw (Micromet, Remet) under water-cooling to obtain 1 mm-thick root slices. Considering the thickness of the blade used for transverse sectioning (approximately 0.3 mm lost each cut), a total of six valuable slices were obtained for each sample and equally divided into coronal (first three slices) and apical portions (last three slices). The push-out test was performed with a plunger with a 1-mm-diameter tip using a universal testing machine (Instron 10/S, MTS) connected to a load cell with a load speed set at 1 mm/min. The loading forces were applied from the apical to the coronal direction. Bond failure was manifested by the dislodgment of the fiber post from the root section. The maximum failure load was recorded in Newtons (N) and converted into Megapascal (MPa) according to the surface adhesion area by dividing the load in Newton by the bonded surface area (SL) in mm$^2$, and SL was calculated as the lateral surface area of a truncated cone using the formula: $S_L = \pi (R + r) \left[ (h^2 + (R - r))^2 \right]^{0.5}$, where $R$ is the coronal post-radius, $r$ the apical post-radius and $h$ root slice thickness. The specimen slices were observed with a stereomicroscope at 40x magnification to determine the failure mode: adhesive, cohesive, and mixed failures.

**Interfacial nanoleakage analysis**

Three additional teeth were prepared for each subgroup (n=3 each, total n=18) and employed for interfacial nanoleakage evaluation, using 1 mm-thick slabs as previously described. Slices were immersed in 50 wt.% ammoniacal silver nitrate solution for 24h in the dark, following the
protocol bt Tay et al.\textsuperscript{8}. The specimens were then thoroughly rinsed in distilled water and immersed in a photo-developing solution for 8 h under a fluorescent light to reduce silver ions into metallic silver grains within voids along the bonded interfaces.

For light microscopy observation, sections were bonded on glass slides, flattened to a thickness of approximately 40 µm under running water with 600-, 800-, 1200- and 2400-grit silicon carbide (SiC) paper (LS2, Remet) and observed under normal transmitted light using a light microscope (Nikon Eclipse). The amount of silver tracer deposited along the bonded interface (the degree of interfacial nanoleakage) was obtained (original magnification: 40X) and scored by two observers on a four-point scale, based on the percentage of the adhesive surface showing silver nitrate deposition, as following: 0: no nanoleakage; 1: < 25% with nanoleakage; 2: 25-50% with nanoleakage: 3: 50-75% with nanoleakage; 4: > 75% with nanoleakage.

A general summary of the protocol is reported in Figure 1.

**Statistical analysis**

For push-out test, as values were normally distributed (Kolmogorov-Smirnov test), data were analyzed using three-way ANOVA test to examine the effects of the factor “cement”, “curing protocol” and “post space region” (apical or coronal) and their interactions on bond strength. Post-hoc pairwise comparisons were performed using the Tukey test.

For nanoleakage analysis, intra-examiner reliability was evaluated using the Cohen’s kappa (κ) statistic. Statistical differences among nanoleakage scores were analyzed with the Chi-square test. Statistical significance was pre-set at p= 0.05.

For all the tests, statistical significance was set at p<0.05. Statistical analysis was performed using STATA 14.0 software (StataCorp). Analysis of the failure modes was conducted using a Chi-square test.
RESULTS

Push out bond strength

Mean bond strength results ± standard deviation, expressed in MPa, are summarized in Table 2 and Table 3. Failure modes, expressed as percentages, are reported in Table 4.

Three-way ANOVA analysis showed that the factors “cement” (p=0.02) and “curing protocol” (p<0.001) had a significant influence on bond strength, while the “post space region” did not influence results (p>0.05). The interaction between factors “cement” and “curing protocol” was significantly affecting the final bond strength (p=0.0047). Pairwise Tukey post-hoc test reported that light curing after 120s since injection showed higher bond strength compared to no light curing and photoactivation after 20s group. No significant differences were reported between SG1 and SG2 (p>0.05). Regarding the type of cement, the Tukey test showed that Bifix SE had a significantly better bond strength than Panavia SA (p<0.05). The failure mode was significantly affected by the resin cement (p<0.005) and the curing protocol (p<0.0001).

Nanoleakage

As Cohen’s kappa (κ) statistic scored 0.83 the agreement with the two observer was classified as almost perfect. Descriptive statistics of interfacial leakage scores are represented in Graph 1. No statistically significant differences were found among the groups in the extent of silver nitrate penetration (p = 0.052), as shown in Figure 2.

DISCUSSION

Based on the present study results, the first null hypothesis is rejected since the formulation of the resin cement significantly affected the bond strength of fiber post.
Previous authors showed product-specific differences in the bond strength of self-adhesive cements inside the root canal\textsuperscript{19,20}, which could be mainly related to their chemical composition. Self-adhesive cements contain conventional mono-, di-, and/or multi-methacrylate monomers, carboxylic or phosphoric acid-functionalized monomers, fillers, and photo-initiators. This unique chemical formulation allows them to combine a curing mechanism based on a free-radical redox polymerization and an acid-base reaction\textsuperscript{21}, which occurs between the acidic functionality on the monomers and the acid-soluble glass or the mineralized tooth surface. Initially, low pH and high hydrophilicity of self-adhesive cements are necessary to obtain adequate wetting and bonding to the tooth substrate. Once adhesion is achieved, the pH increases\textsuperscript{22}, and the material turns more hydrophobic, consequently less susceptible to hydrolysis over time\textsuperscript{23}. The acidity of self-adhesive cements during the initial phase of the polymerization reaction is therefore responsible for smear layer dissolution, dentinal tubules opening, demineralization, and the increase of the micro-porosity of the intertubular dentin necessary for optimal bonding\textsuperscript{24}. Moreover, it has been demonstrated that the neutralization mechanism, which is considered a basic chemical setting process intrinsic to all self-adhesive materials, shows a wide variability among cements\textsuperscript{15,25} and it is of fundamental importance as redox initiators and photo-initiators are sensitive to acidic monomers\textsuperscript{26}. Therefore, considering that Bifix SE has an initial pH of 2.5 and Panavia SA Plus of 4.0, it could be speculated that the intrinsic acidity and a different neutralization process could determine a higher or lower immediate bond strength to root dentin of the tested cements.

The present study results led to reject also the second null hypothesis, since the curing protocol seems to significantly influence the obtained results: a significantly weaker bond strength was observed when tested cements were light-cured after 20s or not cured at all, compared to when photoactivation occurred after 120s. In general, dual-curing self-adhesive
cements possess a complex chemical composition that ensures both chemical- and light-curing, to theoretically fully adhere to the tooth structure, even where light cannot reach. However, some studies suggested that, if not properly light-cured, dual cements result in weaker mechanical properties, which might not be clinically sufficient\textsuperscript{23,25}. It has been recently suggested that the early vitrification induced by insufficient light-activation could interfere with subsequent self-polymerization, compromising the overall curing of dual-cure resin cements\textsuperscript{27}. Thus, immediate light-curing exposure could minimize the extent of the subsequent self-polymerization of the dual-curing cement due to restricted molecular mobility. This mechanism is even more evident when, due to the distance or the interposition of the material between the light source and the dual-curing cement, insufficient curing energy is applied to drive the reaction further the completion\textsuperscript{28}. Thus, it would be better from the standpoint of a degree of conversion to allow the self-curing reaction to progress without additional light exposure\textsuperscript{28}. In this context, it is evident how a waiting period before light activation could significantly increase conversion for the lower dose of light exposed\textsuperscript{29} which is typical for the fiber post luting procedure.

It is well known that dual-curing cements polymer vitrification occurs when the viscosity reaches a high value, reducing the ability of a material to relieve stresses generated by shrinkage\textsuperscript{27,30}. Techniques used to modulate the light-activation are based on the concept that delaying the vitrification allows more relief of shrinkage stress by prolonging the period when the material can flow. A recent study by Pereira \textit{et al.} (2015) noted that a light-curing delay of three minutes significantly reduces shrinkage stress and improves the performance of dual-curing cements\textsuperscript{6}. Considering that the thin push-out test, which is a valid method to analyze fiber post adhesion thanks to the homogeneous stress distribution\textsuperscript{31}, involves the contribution of friction to the detected bond strength values\textsuperscript{32}, it could be speculated that the shrinkage stress has an important
role in the bond strength evaluation of self-adhesive cements. Consequently, a prolonged waiting period before light-activation, above all when light access is difficult, could prolong the pre-gel phase and therefore reduce the initial shrinkage stress along the post space walls. Tay et al. (2005) mathematically calculated that the post space has a C-factor exceeding 200\(^3\). Indeed, the geometry of root canal is complex and may be roughly compared to a very deep class I cavity that has high values of C-factor. Therefore, the fiber post luting procedure represents the worst clinical scenario for a shrinkage stress point of view, and an immediate debonding along the cement/root dentin interface could be easily observed. This is corroborated by the qualitative interfacial nanoleakage analysis performed in the present study, which revealed a smaller amount of silver deposit in samples where 120s waiting time was respected after self-adhesive cement injection. Moreover, a reduced number of adhesive failures was observed when light-curing was later activated, probably suggesting increased friction at the dentin-cement interface.

Based on the present results, the post space area (coronal or apical) did not affect the self-adhesive bond strength to radicular dentin. Despite several previous studies showing differences in bond strength between apical and coronal portions of the root canal\(^3\), some recent findings related to the self-adhesive luting technique were in agreement with the results of the present work\(^3\). Using multi-step adhesive systems, various problems could be encountered that lead to a progressive reduction of the adhesion strength from coronal to apical \(^3\). However, self-adhesive cements rely on micromechanical retention and mainly on the chemical adhesion to hydroxyapatite\(^1\). In the apical area of the post space the dentin is irregular, with fewer tubules filled with minerals, resembling those from peritubular dentin\(^\). There are limited studies regarding the acidity of different regions in the root canal; however, several authors suggest that the apical part is a little more acidic than the cervical part \(^4\). This might influence the bonding
strength in different root canal parts because self-adhesive cement properties highly depend on pH neutralization after curing. Acidity could increase the calcium availability for chemical adhesion, compensating for the effect of light-curing distance, a lower degree of conversion at the apical area. Moreover, self-adhesive cements are more tolerant to dentin moisture variations, whose difficult control in the deep area of the post space can jeopardize dentin bonding with conventional multi-step adhesive techniques. A recent study by Pulido et al. shows that the shrinkage stress of self-adhesive cement is slightly less in the apical third than in the cervical third of the post space.

To conclude, within the limitations of the present in vitro study, the highest bond strength was obtained with photoactivation performed with a 120s delay. Nevertheless, there is no difference in bond strength when a 20s delay before light-curing or no light curing protocols were employed. Finally, GFP cementation resulted to be material dependent.

REFERENCES

Cements and Bond Strength of Fiberglass Post to Root Dentin. Oper Dent 2015;40:E206-221.


FIGURE LEGENDS

Figure 1. General summary of the performed procedures.

Figure 2. Light micrographs showing the adhesive interface created by Panavia SA Plus and Bifix SE of the coronal and apical portions of samples not light-cured and cured after 60s or 120s delay. D = dentin; P = fiber post; pointers = silver nitrate deposits.
Graph 1. Percentages of interfacial nanoleakage expression in resin-dentin interfaces created in radicular dentin with Panavia SA and Bifix SE with different curing protocols (no light, waiting for 20’’ and waiting for 60’’) for the coronal and the apical root area. Interfacial nanoleakage was scored based on the percentage of the adhesive surface showing silver nitrate deposition: 0, no nanoleakage; 1, <25% with nanoleakage, 2, 25% to ≤50% with nanoleakage; 3, 50% to ≤75% with nanoleakage; 4, >75% with nanoleakage. Increasing grey shades indicate increasing interfacial nanoleakage expression.
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<th>Material</th>
<th>General description</th>
<th>Manufacturer</th>
<th>Composition</th>
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<tr>
<td><strong>PANAVIA SA Cement</strong></td>
<td>Universal self-adhesive, dual cured resin cement</td>
<td>Kuraray, Tokyo, Japan</td>
<td>Paste A: 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Bisphenol A diglycidylmethacrylate (Bis-GMA) Triethyleneglycol dimethacrylate (TEGDMA) Hydrophobic aromatic dimethacrylate 2-Hydroxymethacrylate (HEMA), Silanated barium glass filler, Silanated colloidal silica, dl-Camphorquinone, Peroxide, Catalysts, Pigments Paste B: Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Silanated barium glass filler, Surface treated sodium fluoride Accelerators, Pigments</td>
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<tr>
<td><strong>Bifix SE</strong></td>
<td>Universal self-adhesive, dual cured resin cement</td>
<td>Voco, Cuxhaven, Germany</td>
<td>Base: Bis-GMA, UDMA, Acidic phosphate monomers, Glycerindimethacrylate, Benzoylperoxide, Aerosol silica, Hydroxypropylmethacrylate. Catalysts, Initiators, Stabilizers, Glassfillers(70wt%) Catalyst: UDMA, Glycerindimethacrylate, Catalysts, Initiators</td>
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<tr>
<td><strong>Rebilda Post #15</strong></td>
<td>Glass fiber reinforced post</td>
<td>Voco, Cuxhaven, Germany</td>
<td>Solid composite of glass fibers, inorganic fillers, PDMA</td>
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*Table 1. General description of materials used in the present study.*
Table 2. Mean bond strength (MPa± standard deviation) of the two different cements for the coronal portion of the root. Different superscript upper case letters indicate differences within the same column (p<0.05), different superscript lower case letters indicate differences within the same row (p<0.05).
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<td>Light after 20&quot; (SG2)</td>
<td>Light after 120&quot; (SG3)</td>
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<tr>
<td>Panavia SA (G1)</td>
<td>8.87\text{A}_a ± 5.05\text{A}_a</td>
<td>7.43\text{A}_a ± 3.14</td>
<td>11.65\text{A}_a ± 3.55</td>
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<td>Bifix SE (G2)</td>
<td>6.14\text{A}_b ± 2.77\text{A}_a</td>
<td>10.03\text{A}_ab ± 3.44</td>
<td>14.56\text{A}_a ± 5.69</td>
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Table 3. Mean bond strength (MPa± standard deviation) of the two different cement for the apical portion of the root. Different superscript upper case letters indicate differences within the same column (p<0.05), different superscript lower case letters indicate differences within the same row (p<0.05).
Table 4. Failure modes, expressed as percentages, of the investigated self-adhesive cements with respect to post space region, employed cement and waiting time before light curing (adh = adhesive; coh = cohesive; mix = mixed).

<table>
<thead>
<tr>
<th>Cement</th>
<th>No light (SG1)</th>
<th>Light after 20” (SG2)</th>
<th>Light after 120” (SG3)</th>
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<tr>
<td></td>
<td>Adh</td>
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