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Hydrolytic degradation of silorane- and methacrylate-based composite restorations: Evaluation of push-out strength and marginal adaptation

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Abstract

Objectives. This study compared the hydrolytic degradation of composite restorations based on methacrylate and silorane systems regarding bond strength and marginal adaptation. ***Materials and methods.*** Sixty bovine incisors were ground flat to obtain a 2-mm thick slice in which conical preparations were made. The specimens were randomly distributed into four groups ($n = 15$) according to the restorative system (silorane–Filtek LS/P90 adhesive; methacrylate–Filtek P60/Adper Easy Bond) and the degradation protocol (control: immediate evaluation; hydrolytic degradation: 6 months storage in water at 37°C). Marginal adaptation was evaluated using a dye staining technique. Digital images of the stained gaps were obtained to calculate the marginal gap (%), the ratio between the stained margins and the total length of the margin. Push-out bond strength test was conducted (0.5 mm/min). Marginal adaptation data was submitted to Kruskal-Wallis test and the bond strength data to two-way ANOVA/Tukey's test ($\alpha = 0.05$). ***Results.*** The marginal adaptation was neither affected by the restorative system nor by the degradation protocol, although the number of perfect sealed reduced after 6 months. No significance was observed among the groups. No significance was noted between the silorane- and the methacrylate-based restorations for immediate bond strength. After the hydrolytic degradation, the silorane system showed higher bond strength then the methacrylate restorations. ***Conclusion.*** The silorane and methacrylate restorative systems produce restorations with similar immediate interfacial quality and 6 months of water storage does not cause significant bonding degradation for both systems. The silorane restorations show an increase in the bond strength after 6 months.

Key Words: *composite resins, dental bonding, dental marginal adaptation, methacrylate, silorane*

Introduction

Recently, in an attempt to reduce shrinkage stress, a new classification of resin composites known as low-shrinkage composites was developed. In this way, a silorane-based composite was introduced containing cationic ring-opening monomers: a compensating mechanism for shrinkage stress achieved during polymerization [1]. This monomer system was obtained from the reaction of oxirane and siloxane molecules [1]. This resin composite presents low polymerization shrinkage due to the ring-opening oxirane and an increased hydrophobicity due to the presence of the siloxane. The silorane-based composite revealed a decreased water sorption, solubility and associated diffusion coefficient compared to those observed when methacrylate-based composites were tested [1]. In a previous study [2], it was found that the cusp deflection caused by polymerization shrinkage was significantly lower when extracted premolars were restored with silorane material in comparison to that noted when a methacrylate-based composite was applied. In addition, no microleakage was found when Class II MOD preparations were restored with a silorane-based composite [3].

The silorane restorative system contains a dedicated adhesive system designed to link the hydrophilic dentin and hydrophobic silorane composite. P90 primer and bond are presented in separate bottles and are polymerized as separate layers. According to the manufacturer, P90 primer presents a pH 2.7, providing a mild etching and demineralization of

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the tooth structure. A recent study proved that P90 primer has also been claimed to present chemical bonding to the hydroxyapatite crystals [4]. A previous study supported the hypothesis that an optimal stability of silorane adhesive can be achieved; however, concerns have been reported over the quality and long-term stability of the hybrid layer created by applying the P90 adhesive system [5].

This study evaluated the push out bond strength and the marginal adaptation of rounded restorations filled with different posterior restorative systems (a silorane-based or a methacrylate-based system). The results of both parameters after 6 months of water storage were compared to that obtained immediately (after 24 h). The following research hypotheses tested were: (1) the bond strength and marginal sealing of restorations filled with a silorane-based restorative system will be superior to that observed for restorations filled with a methacrylate-based system; (2) hydrolytic degradation will impact the bond strength and the marginal sealing of composite restorations for both methacrylate- and silorane-based restorative systems after 6 months.

Methodology

Experimental design

In this *in vitro* study, marginal adaptation and bond strength of restorations were evaluated according to the factors: (1) Restorative system at two levels: methacrylate-based (Filtek P60/EB adhesive) and silorane-based (Filtek LS/P90 adhesive system); and (2) degradation protocol at two levels: control (24 h after restorative procedure) and hydrolytic degradation (6 months storage in water at 37°C). Four groups (n = 15) were obtained by the product among factors under study (P60-EB/24 h, P60/6 months; LS-P90/24 h and P90/6 months).

Preparation of specimens

Sixty bovine incisors were selected, cleaned and stored in a 0.5% Chloramine T solution at 4°C for no more than 1 week. The crowns were cut-off in the cement–enamel junction (CEJ) using a water-cooled rotating diamond wheel (Isomet, Buehler Ltd., Evanston, IL). Then, the crowns were ground flat to obtain 2-mm thick slices in which a conical preparation was made with margins in dentin (top diameter of 4.0 mm, bottom diameter of 3.0 mm and 2.0 mm thick) using a diamond tip (#3131; Brasseler, Savannah, GA), mounted in a high-speed hand piece, under constant air-water cooling in a standard cavity preparation appliance. In the push out bond strength test, the compressive force of the probe is applied at the bottom surface of the restoration in order to induce the rupture of the tooth-composite bonding area. The C-factor of the preparation was 1.15 and the volume of composite was ~ 19 mm^3. The diamond burs were replaced after every five preparations.

For preparations that received the Adper Easy One adhesive (P60 Groups), it was rubbed onto the preparation surfaces for 20 s, followed by a gentle air blowing for 5 s. Adhesive was then also polymerized for 10 s. For preparations that received the silorane-based composite (P90 Groups), a two-step, self-etch adhesive system (P90 System adhesive, 3M ESPE, St. Paul, MN) was previously applied to all aspects of the preparation, also following manufacturer's instructions. P90 primer was applied for 15 s with a microbrush, followed by gentle air dispersion and 10 s of light curing. P90 bond was also applied with a microbrush, followed by gentle air dispersion and photoactivation for 10 s. The characteristics of the resin composites as well as the dentin bonding adhesive systems selected are described in Table I. All restorative materials were photoactivated using a LED light-curing unit (Bluephase, Ivoclar-Vivadent, Schaan, Liechtenstein) with a power density of 1200 mW/cm^2. Prior to testing and throughout the experiment, the power density was monitored using a handheld radiometer (Model 100, Demetron Research Corp., Danbury, CT). Resin composites (shade A3) were applied in a bulk increment and photoactivated with the distal end of the light-curing tip perpendicularly placed within 0.1 mm of the occlusal surface of the restoration [6]. The specimens were then stored at 37°C in physiological saline solution for 24 h. Restorations were then finished and polished using 8- and 16-blade carbide burs (ET4 and ET3F, Brasseler, Savannah, GA, respectively) and abrasive discs (Sof-Lex, 3M ESPE, St. Paul, MN) mounted in a slow-speed hand-piece. The specimens were used for both the marginal adaptation evaluation and for the push-out bond strength test.

Marginal adaptation evaluation

After the storage time, a dye staining technique was performed [7,8] for assessing gap formation in the internal margins of resin composite restorations. This method demonstrated a strong correlation with the SEM analysis using the replica technique, which is considered the gold standard method for assessing gaps in composite restoration. In this way, in order to assess the gap formation, the specimens were air-dried and a 1% acid red propylene glycol solution (Caries Detector, Kuraray, Osaka, Japan) was applied on the margins of the restorations using a microbrush for 20 s [7]. After the dye staining, specimens were water-rinsed and air-dried and digital images were obtained. The length of stained marginal interface was measured using the Image Tool 2.0 software (UTHSC, San Antonio, TX). Marginal gap (%) was calculated as the ratio of the stained margins to

Table I. Materials used in the study.

Materials	Composition	Lot #
Filtek LS (LS) (3M ESPE)	3,4-Epoxycyclohexylethylcyclopolymethylsiloxane, bis-3,4-poxycyclohexylethylphenylmethylsilane;	9ER
	Silanized quartz; yttrium fluoride; 76wt%.	
Filtek P60 (P60) (3M ESPE)	Bis-GMA; Bis-EMA; UDMA; TEGDMA;	9PG
	Silica nanofiller; 83wt%.	
P90 System Adhesive (P90) (3M ESPE)	Two-bottle self-etch adhesive system;	9BH (B)
	Primer: phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers.	9BL (P)
	Bond: hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silane-treated silica filler, initiators, stabilizers.	
Adper Easy Bond (EB) (3M ESPE)	One-step, self-etching adhesive system;	
	Bis-GMA; polyalkenoic acid co-polymer; dimethacrylates; phosphorylated methacrylates;	9WF
	HEMA; photoinitiators; ethanol; water; nanofiller particles.	

Bis-GMA, bisphenol-glycidyl-methacrylate; Bis-EMA, bisphenol-a-ethoxy dimethacrylate; UDMA, urethane-dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; HEMA, hydroxyethyl methacrylate.
Note: The brand name Filtek LS is used in other countries as Filtek Silorane and Filtek P90.

the total length of the margin. Also, the number of perfectly sealed restorations was recorded. The non-parametric Kruskal-Wallis test (5% of significance) was selected based on the previous non-normal distribution detected from the Kolmogorov-Smirnov and Shapiro-Wilk tests.

Push-out bond strength test

After evaluation of the marginal adaptation, the bond strength was conducted by using a push out bond strength test. An acrylic device with a central hole was adapted at the base of a Universal Testing machine (Instron Model 4411, Buckinghamshire, UK). The central hole was used in order to place the specimen with its preparation bottom (the smaller diameter of the restoration) sided up (Figure 1). A rounded probe was adapted to the testing machine and a compressive force was applied to the bottom surface of the restoration in order to induce the rupture of the tooth-composite bonding area (Figure 1). The speed at which the push out test was conducted was 0.5 mm/min. The results (in Kgf) were divided by the bonded area (22.65 mm^2) and transformed in

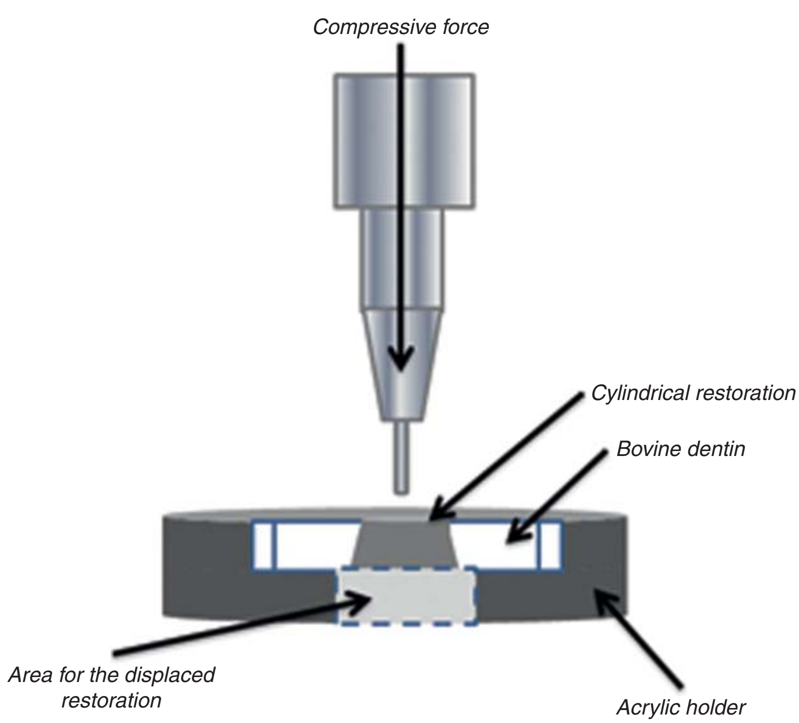

Figure 1. Schematic representation of the push-out bond strength test.

MPa. The results of push out bond strength test were submitted to 2-way ANOVA (factors: restorative system and evaluation time) and Tukey's tests (5% of significance).

Failure mode analysis

After the test, the analysis of the failure modes was performed by using a dissecting microscope (Stereozoom, Bausch & Lomb, Rochester, NY), with the classification, as follows:

- Type 1: Adhesive;
- Type 2: Mixed 1: adhesive/cohesive in the composite;
- Type 3: Mixed 2: adhesive/cohesive in the composite and dentin;
- Type 4: Cohesive of the composite; and
- Type 5: Cohesive in dentin.

Results

The mean percentages of gap formation are presented in the Table II. In the Figure 2, the incidences of the failure modes are displayed. The relative incidence of specimens with perfect seal x presence of gaps are presented in the Figure 3. According to Table II, no significance was noted among the experimental groups in terms of gap formation (power of the test = 82.16%). Despite the decrease in the incidence of perfect sealed margins after 6 months of water storage, all of the experimental groups exhibited low percentages of gaps (Figure 3).

The results of the push-out bond strength test are described in Table III. No significance was noted between the restorative systems P60 and P90 when the specimens were immediately evaluated (24 h). However, after 6 months of water storage, the bond strength of the preparations filled with the P90 restorative system presented a significantly higher bond strength mean in comparison to that of preparations filled with the P60 restorative system. In addition, when the means of both evaluation times were compared, a significant increase in the bond strength mean was noted after 6 months; for the P60 restorative system no significance was noted after 6 months when compared to that observed after 24 h. The highest push out strength mean was noted for the P90 restorative system after 6 months.

The restorative system P60 revealed most incidences of type 3 mode of failure (mixed 2: adhesive/cohesive in the composite and dentin), whereas an increase in the adhesive mode of failure (type 1) was observed after 6 months (Figure 2). For P90 restorative systems, an increase in type 1 (adhesive failure) occurred with time (from ~ 15–50%). The type 2 failure mode (mixed 1: adhesive/cohesive in the composite) was frequent in the 24 h evaluation time (35%) but decreased to ~ 5% after 6 months (Figure 2).

Table II. Gap (%) means according to the restorative system and the evaluation time.

	Evaluation time	
Restorative system	24 h	6 months
Adper Easy Bond/Filtek P60	0.28^{A}	0.07^{A}
P90 System Adhesive/Filtek LS	0.35^{A}	0.21^{A}

Similar capital letters: not significant ($p > 0.05$).

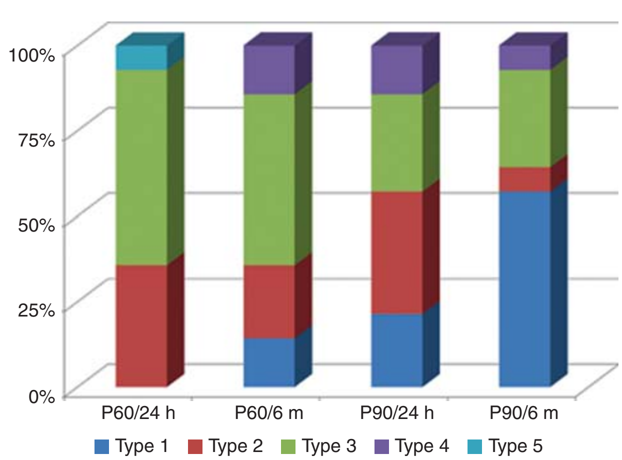

Figure 2. Incidence of failure modes (%).

Discussion

The first hypothesis tested, that the bond strength and marginal sealing of restorations filled with silorane-based restorative system would be superior to that observed for restorations filled with a methacrylate-based system, was not accepted. No significance was noted when the bond strength and marginal sealing of

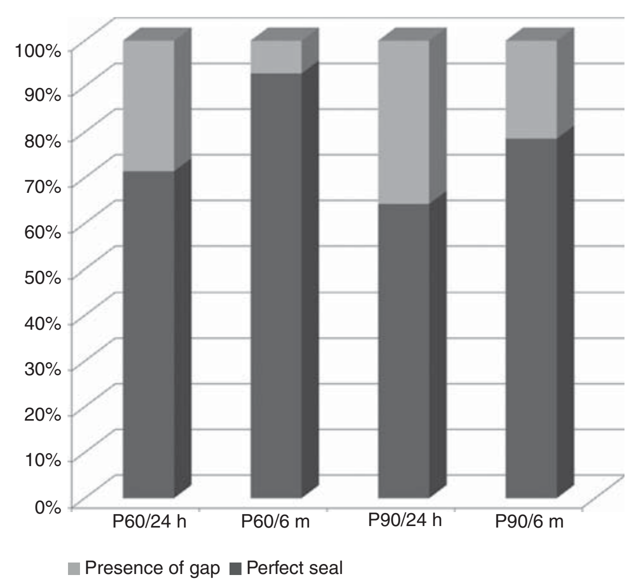

Figure 3. Percentage (%) of gap-free interfaces according to the restorative systems and evaluation times.

Table III. Push out strength means according to the restorative system and the evaluation times.

	Evaluation time	
Restorative system	24h	6 months
Adper Easy Bond/Filtek P60	8.0 (1.4)[aA]	9.6 (4.4)[bA]
P90 System Adhesive/Filtek LS	9.8 (2.3)[aB]	12.5 (3.8)[aA]

Mean (SD) values followed by different capital letters in row and small letters in column: significant ($p < 0.05$).

restorations filled with silorane-based restorative systems were compared to that of with a methacrylate-based system. Previous studies have reported contradicting results when testing the low-shrinkage silorane composite in comparison to methacrylate-based composites. A previous study [9] found that the shrinkage stress produced by silorane-based composites was comparable to that seen in some methacrylate-based composites. The authors pointed out that reduced polymerization shrinkage is not a guarantee of reduced stress development in composites. These results were confirmed in another study in which no differences in terms of gap formation were found when a silorane restorative system was compared to a nanohybrid methacrylate-based one in Class V preparations [10]. In another study [11], it was found that the silorane material presented better behavior than the dimethacrylate materials when marginal adaptation was compared to standardized cylindrical preparations in dentin. On the other hand, a clinical study [12] revealed that the excellent results exhibited by a silorane-based composite in laboratory tests were not clinically validated.

In the present study, self-etching systems were used as bonding approaches in all of the experimental groups. The P90 bond comprises a viscous adhesive component which contains a hydrophobic bifunctional methacrylate-based monomer compatible with the hydrophobic silorane composite. On the other hand, the P90 primer contains phosphorylated methacrylates and copolymers with carboxylic acid groups contained in a variety of resin-modified glass ionomers. It also contains co-monomers like BisGMA and HEMA, dissolved in a mixture of water/ethanol solvent. Unlike any other two-steps, in which the primer and the bond components are applied and mixed on the dentin surface before curing, the P90 primer and bond are separately applied and photoactivated as separate layers. In spite of the combination of acidic monomers and water/ethanol solvents, according to the manufacturer, special care was taken in order to obtain a stable formulation. According to manufacturer's information, Adper Easy One resembles the formulation of the adhesive Adper Single Bond 2, with the exception of the presence of methacrylated phosphoric esters in the former. This modification in the adhesive composition reduced the pH (from 4.3 to 3.5) and dispensed the application of the aggressive, low-pH phosphoric acid gel (pH 0.6), increasing the ability of the adhesive to effectively etch and permeate the smear layer. This somehow explains the reasons why the bond strength and the gap formation were similar for both experimental groups in the 24 h evaluation time.

The interface is considered the weakest area of bonded restorations. Marginal discolorations, poor marginal adaptation and subsequent loss of retention of the restoration are claimed to be the most frequent occurrence [13]. Hydrolysis, a chemical process that breaks covalent bonds between the polymers, is considered as one of the main reasons for resin degradation within the hybrid layer [14,15], as it contributes to the reduction in the bond strength over time [16]. In spite of the excellent immediate and short-term bonding effectiveness of dental adhesives, the longevity of resin-bonded interfaces is questionable. It has been claimed that the immediate bonding to dentin frequently does not correlate with longevity over time as a variety of signs of degradation are detected even in short periods (i.e. 6 months) [14]. In the present study, the bond strength means and the marginal sealing were not impacted by the hydrolytic degradation. In spite of the statistical equivalence in terms of bond strength at the 24 h evaluation time, significantly higher bond strength mean was noted for the P90 restorative system after 6 months, in comparison to that of the P60 system. The second research hypothesis, which anticipated that the hydrolytic degradation would decrease the bond strength and impact the marginal sealing of composite restorations for both the methacrylate- and the silorane-based restorative systems after 6 months, was not accepted.

Reasons that explain these results rely on the characteristic of the methodology and of the specimens tested. Most studies evaluating the hydrolytic degradation of the adhesive interface are conducted by using the microtensile bond strength test, which allows an acceleration of the degradation process due to the reduced dimensions of the stick-shaped specimens. In that sense, it is also important to evaluate bonding degradation using a model in which the restoration is entirely evaluated as observed in the push-out test. It has been pointed out that the push-out test results can be used to establish a relationship between the applied load and the fracture/failure behavior of the restorative system interfaces [17]. When a compressive load is applied to the top of the composite restoration, shear stresses are introduced at the interface with maximum value occurring at the region near the top face of the cylindrical restoration [17].

Reasons that explain the significant increase in the bond strength of the silorane restorative system can be attributed to two main factors: the polymerization in silorane systems occurs more slowly (which may interfere with the 24 h results) and the presence of a highly hydrophobic layer in the P90 adhesive system would allow the preservation of the bonding interface in face of the hydrolytic degradation. The slow curing of silorane composite has been reported [2,18]. It has been claimed that the slow curing may cause a delay in the development of the mechanical properties of the silorane material. These findings were corroborated in the present study when evaluating the failure mode in which a high incidence of cohesive failure in the composite was observed (types 2, 3 and 4) at the 24 h evaluation time (Figure 2). Additionally, both adhesive systems present the Vitrebond copolymer with its carboxylic acid functionality used in a variety of resin-modified glass ionomers. In the adhesive Adper Easy Bond, the phosphoric esters and the Vitrebond copolymer form a chemical bond to the hydroxyapatite by forming a complex with the calcium ions. The silorane primer contains phosphorylated methacrylates, as well as adhesives for adhesion to enamel and dentin. The P90 primer has also been claimed to present chemical bonding to the hydroxyapatite crystals, already confirmed in a recent study [19]. In this way, due to the presence of this ionomer compound, the maturation of the bonding may take more than 24 h to consolidate.

The increase in the push out bond strength observed after 6 months can also be explained by the silorane bond chemistry. The silorane adhesive system includes the P90 bond component that includes a hydrophobic monomer that bridges with the silorane composite. It somehow may have contributed to the preservation of the bonding interface as well. Bonding degradation has been correlated with adhesive hydrophilicity and water sorption: the higher the hydrophilicity, the higher the hydrolytic degradation. However, it must be pointed out that this adhesive interface of the silorane restorative system is vulnerable. A previous study [20] demonstrated an intermediate zone of ~ 1 μm between the silorane primer and the bond detected by using Micro-Raman spectroscopy, which somehow explains the higher incidence of adhesive failures in the silorane groups. According to these authors, this may be the weakest link in the failure mechanism of silorane restorations and certainly needs further investigations. Also, it has been pointed out that there may be a difference in the chemistry between the silorane composite and the adhesive that connects it with the tooth structure, allowing failures between the bond and the composite [21]. It also contributes to explain the high incidence of adhesive failures noted in the silorane restorations.

Regarding the methacrylate-based restorative system, although a simplified one bottle adhesive system was used, no reduction in the bond strength and an impact in the marginal adaptation were observed. Considering these results, it could be speculated that the reported degradation of bond strength observed in microtensile bond strength tests where sticks are stored in water for several months can be over-estimated as, clinically, the degradation mechanism in a restoration occurs slowly, even when simplified adhesive systems are applied.

The present study evaluated the bonding effectiveness and the marginal integrity of a silorane restorative system to dentin in comparison to that of a methacrylate-based one. It has been pointed out that a synergic effect, a low-shrinking composite/ an effective adhesive system in the silorane restorative system allowed an improvement in the bonding to dentin over time [22]. It was found that the immersion in water for 6 months caused no reduction in the bond strength, although a reduction in the perfect sealed margins was noted. This is a critical clinical issue in adhesive dentistry and has to be further substantiated by evaluating the longevity of the restorations. Extrapolations to clinically support and validate the results using different adhesive system categories and the silorane composite need to be done with caution, since future studies are required.

Conclusion

The present study deals with a clinically relevant issue, which can influence the dental practice. Within the limitations of this study, it can be concluded that:

- Hydrolytic degradation causes no effects in the push-out bond strength and in the marginal adaptation after 6 months of water storage, despite the reduction in the percentage of perfect sealed margins;
- Silorane- and methacrylate-based restorations present similar immediate push-out bond strength and marginal adaptation; and
- Silorane-based restorations present an increase in the push-out bond strength mean after 6 months of water storage.

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