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Impact of simplified chemical treatment of semi-direct restorations cemented with self-adhesive resin cement on dentin bond durability and nanoleakage

Impacto do tratamento químico simplificado de restaurações semidiretas cimentadas com cimento resinoso autoadesivo na durabilidade da união à dentina e nanoinfiltração

Impacto del tratamiento químico simplificado de las restauraciones semidirectas cementadas con cemento de resina autoadhesivo sobre la durabilidad de la unión unión a la dentina y la nanofiltración

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Abstract

The present study aimed to evaluate the impact of different chemical surface treatments and aging on dentin bond strength and nanoleakage of semi-direct restorations cemented with self-adhesive resin cement. One hundred and sixty semi-direct composite restorations (4.8 x 2.8 x 4.0 mm) were produced to fill dentin preparations in bovine tooth, and the specimens were divided into 8 groups according to chemical treatment [No treatment, Silane (S), Scotchbond™ Universal adhesive (SBU), and S+SBU], and aging time in water [24 h and 6 months]. Push-out bond strength (PBS) was measured through a universal testing machine (1.0 mm/min), failure modes by a dissecting microscope, and nanoleakage by scanning electron microscopy. Two-way ANOVA and Tukey's post-hoc ($p<0.05$) test were used to analyze PBS data, whilst failure modes and nanoleakage were analyzed descriptively. The bond durability was influenced by different chemical surface treatments after 6 months of aging in distilled water, with the best performance for the group that uses silane associated with the universal adhesive. Nanoleakage was greater at the dentin-cement interface, from the base of the restorations. However, the infiltration of silver crystals at the cement-resin interface was not influenced by the different chemical treatments applied. The results of the study suggest that self-adhesive cement promotes efficient adhesion to the interface that improves over time without the need for chemical surface treatment or when using SBU, with or without silane.

Keywords: Adhesiveness; Resin cements; Dental permanent filling; Dental restoration failure; Longevity.

Resumo

O presente estudo teve como objetivo avaliar o impacto de diferentes tratamentos químicos de superfície e envelhecimento na resistência de união à dentina e nanoinfiltração de restaurações semidiretas cimentadas com cimento resinoso autoadesivo. Cento e sessenta restaurações semi-diretas (4,8 x 2,8 x 4,0 mm) foram produzidas para preencher preparos em dente bovino, e os espécimes foram divididos em 8 grupos de acordo com o tratamento químico [Sem tratamento, Silano (S), Scotchbond ™ Adesivo universal (SBU) e S + SBU], e tempo de envelhecimento em água [24 h e 6 meses]. A força de união por push-out (PBS) foi medida por meio de uma máquina de ensaio universal (1,0 mm / min), os modos de falha por um microscópio de dissecação e a nanoinfiltração por microscopia eletrônica de varredura. ANOVA de dois fatores e teste post-hoc de Tukey ($p <0,05$) foram usados para analisar os dados de PBS, enquanto os modos de falha e nanoinfiltração foram analisados descritivamente. A durabilidade da união à dentina foi influenciada por diferentes tratamentos químicos de superfície após 6 meses de envelhecimento em água destilada, com melhor desempenho para o grupo que utiliza silano associado ao adesivo universal. A nanoinfiltração foi maior na base das restaurações, na interface dentina-cimento. No entanto, a infiltração dos cristais de prata na interface cimento-resina não foi influenciada pelos diferentes tratamentos químicos aplicados. Os resultados do estudo sugerem que o cimento autoadesivo promove uma adesão eficiente à interface que melhora com o tempo, sem a necessidade de tratamento químico de superfície ou quando se utiliza SBU, com ou sem silano.

Palavras-chave: Adesividade; Cimentos de resina; Restauração dentária permanente; Falha de restauração dentária; Longevidade.

Resumen

El presente estudio tuvo como objetivo evaluar el impacto de diferentes tratamientos químicos de superficie y el envejecimiento en la fuerza de unión de la dentina y la nanofiltración de restauraciones semidirectas cementadas con cemento de resina autoadhesivo. Se produjeron ciento sesenta restauraciones de composite semidirectas (4,8 x 2,8 x 4,0 mm) para rellenar preparaciones de dentina en diente bovino, y las muestras se dividieron en 8 grupos según el tratamiento químico [Sin tratamiento, Silano (S), Scotchbond ™ Adhesivo universal (SBU), y S + SBU] y tiempo de envejecimiento en agua [24 hy 6 meses]. La fuerza de unión por expulsión (PBS) se midió a través de una máquina de prueba universal (1,0 mm / min), los modos de falla con un microscopio de disección y la nanofiltración mediante microscopía electrónica de barrido. Se utilizaron ANOVA bidireccional y la prueba post-hoc de Tukey ($p < 0.05$) para analizar los datos de PBS, mientras que los modos de falla y la nanofiltración se analizaron descriptivamente. La durabilidad de la unión fue influenciada por diferentes tratamientos químicos de la superficie luego de 6 meses de envejecimiento en agua destilada, con el mejor desempeño para el grupo que usa silano asociado al adhesivo universal. La nanofiltración fue mayor en la interfase dentina-cemento, desde la base de las restauraciones. Sin embargo, la infiltración de cristales de plata en la interfase cemento-resina no fue influenciada por los diferentes tratamientos químicos aplicados. Los resultados del estudio sugieren que el cemento autoadhesivo promueve una adhesión eficiente a la interfase que mejora con el tiempo sin necesidad de tratamiento químico de la superficie o cuando se usa SBU, con o sin silano.

Palabras clave: Adhesividad; Cementos de resina; Restauración dental permanente; Fallo de la restauración dental; Longevidad.

1. Introduction

To minimize technical difficulties in the use of resin composite by the direct technique, an indirect technique allows for better occlusal anatomy, proximal contact, and better marginal adaptation due to the lower stresses caused by the polymerization contraction. However, when opting for an indirect restoration, it is necessary to use a temporary restoration and the contribution of a laboratory technician that leads to increased costs and treatment time (Rho, et al., 2013; Alharbi, et al., 2014).

In this way, the semi-direct technique can be an excellent treatment option for large posterior restorations since it adds the advantages of direct and indirect techniques such as decreased polymerization stresses in the adhesive interface, aesthetic refinement, and adjustments outside the oral cavity, precision of restoration margins, low operational cost and time of execution (Bandéca, et al., 2012; Fahl Jr, 2015).

One of the main factors for clinical success in cemented restorations is the luting technique, as this will determine the strength and adhesion at the tooth-restoration interface to favor the bonding durability in restorations (Sokolowski, et al., 2018). For making semi-direct restorations on posterior teeth, ceramics or composite resins can be used. Conditioning with hydrofluoric acid followed by the application of silane is the most used method for the surface treatment of ceramics since the acid promotes a mechanical overlap and silane a chemical bonding between the ceramic and the resin (Hooshmand, et al., 2012). However, in clinical practice, the use of silane as a bonding agent for indirect restorations can cause problems in adaptation, because as the oral environment is humid, it causes its degradation over time (Martinlinna, et al., 2018).

As a result, other strategies have been studied to improve the bond strength between indirect restoration and resin cement, such as the use of universal adhesives that propose a new approach, especially those that already contain silane in their composition, simplifying the adhesion protocol and, consequently, reducing the clinical time (Kim, et al., 2015; Xie, et al., 2016). Even so, the application of intermediate agents results in a multiphase layer, which can introduce flaws between the applications of the materials, making the interface more susceptible to infiltration (Papacchini, et al., 2007a).

Self-adhesive cements were developed to simplify luting procedures by reducing the number of luting steps, making the luting process simpler and faster (Skupien, et al., 2015). Because they do not require preparation of the substrate, the performance of self-adhesive cements appears to be more dependent on the material than on the technique itself (Almeida, et al., 2018).

Therefore, little is known about the effectiveness of surface treatments as intermediate agents for luting semi-direct restorations with self-adhesive cement and there is still no established luting protocol in the literature for this type of restoration. The objective of this in vitro study was to evaluate the effect of different chemical treatments and aging on dentin bond durability and nanoleakage of semi-direct restorations cemented with self-adhesive cement. The null hypothesis was that dentin bond durability and nanoleakage of semi-direct restorations would not be influenced by the tested chemical treatments and aging times.

2. Material and Methods

2.1 Experimental design

This in vitro study involved a 4 x 2 factorial design. Four chemical treatments (no treatment, Silane, Scotchbond™ Universal adhesive (SBU), and Silane (S) + SBU – 3M ESPE, St. Paul, MN, USA), and two aging times in water (24 h and 6 months) were tested. The response variables were: Bond Strength (BS), Failure Mode (FM), and Nanoleakage (NL). The materials used in the study are described in Table 1.

Table 1: Description of the materials used in the study.

Product	Manufacturer	Composition^a	Lot
Filtek Z250XT A2	3M ESPE, St. Paul, MN, USA	Silane treated ceramic, bisphenol a diglycidyl ether Dimethacrylate (BisGMA), bisphenol a polyethylene glycol, Diether dimethacrylate diurethane dimethacrylate (UDMA), silane treated silica.	1817200561
RelyX U200 A2	3M ESPE, St. Paul, MN, USA	Base paste: silane treated glass powder, substituted dimethacrylate,Silane treated silica, sodium p-toluenesulfinate, 1-benzyl-5-phenyl-barbic-acid, calcium salt, 1,12-dodecane dimethycrylate, calcium hydroxide; Catalyst paste: silane treated	1727700745

		glass powder, 2-propenoic acid, 2-methyl-, 1,1'-[1-(hydroxymethyl)-1,2-Ethanediyl] ester, reaction products with 2-hydroxy-1,3-Propanediyl dimethacrylate and phosphorus oxide, Triethylene glycol dimethacrylate (TEGDMA), silane treated silica, glass powder, Sodium persulfate, Tert-butyl peroxy-3,5,5-trimethylhexanoate.	
Scotchbond Universal	3M ESPE, St. Paul, MN, USA	Bis-GMA, 10-MDP, dimethacrylate resins, HEMA, copolymer of acrylic and itaconic acids, silane-treated silica, ethanol, water, initiators, and silane.	2508418
RelyX Ceramic Primer	3M ESPE, St. Paul, MN, USA	Methacryloxypropyltrimethoxysilane, ethanol and water.	1813400582

10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: bisphenol A glycidylmethacrylate; HEMA: 2-hydroxyethyl methacrylate; TEGDMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate.

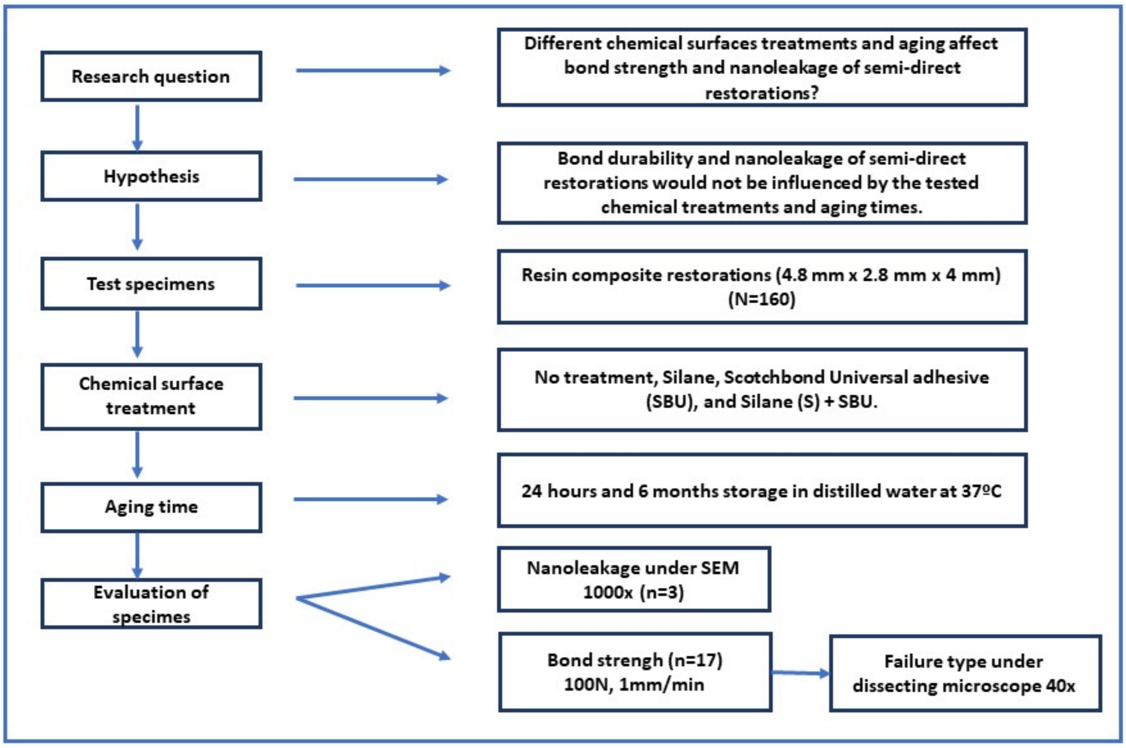
[a] *Data supplied by the manufacturer.*

Source: Personal archives, (2020).

2.2 Specimen preparation and cementation

Bovine incisors (n = 160) without enamel cracks or structural defects were selected and disinfected in an aqueous solution of thymol (0.1%) at 37 ºC for one week. The roots were removed by cutting at the cementoenamel junction (CEJ) with a water-cooled diamond disc (Dentorium, New York, USA) coupled to a precision cutting machine (Dentscler, SP, Brazil). The crowns were cut crosswise 4 mm from the edge of the CEJ to obtain a 4.0-mm-thick disc with the pulp cavity in the center. The top and bottom dentin surfaces of the samples were sanded flat with 400 and 600 granulation discs (Labopol-21, Copenhagen, Denmark). Standardized conical cavities (4.8 mm top diameter x 2.8 mm bottom diameter x 4 mm height) were prepared with Maxicut burs (Komet Inc, Lemgo, Germany) using a handpiece motor under air-water cooling. The bur was replaced every 10 preparations (Souza-Lima, et al., 2017). The preparations were filled with a composite resin (Filtek Z250 XT A2, 3M) by 2-mm increments that were each light-cured separately for 20 s (Coltolux® LED, Coltène, Altstätten, Switzerland – 1264 mW/cm2). The fillings were removed from the preparations and heat-cured in a microwave oven (Consul, SC, Brazil), for 5 minutes at power 800 W2 (Godoy, et al., 2014). The top and bottom surfaces were finished/polished with #600 to #1200 grit paper discs. The chemical treatments were applied to the surface of the restoration with a flexible rod by scrubbing for 20 seconds, followed by volatilization for 5 seconds. Soon after, the restoration was cemented in the preparation. The cement was dispensed on a glass plate, mixed, and applied on the preparation walls and restoration edges. The restoration was placed in the cavity, the excess was removed with an exploratory probe and flexible rod, and light-cured for 20 seconds. Then, half of the specimens were stored 24 and a half for 6 months in water before bond strength, failure mode, and nanoleakage analyses. The entire protocol used in the study is found in the flowchart (Figure 1).

Figure 1: Flowchart of the study protocol.



S: silane; SBU: Scotchbond Universal; SEM: Scanning Electron Microscopy.
Source: Personal archives, (2020).

2.3 Push-out bond strength and failure modes

To measure the bond strength, the push-out extrusion test was used in a universal testing machine (Microtensile OM150, Odeme, Luzerna, SC, Brazil) after 24 h. The specimens (n = 17) were placed with the bottom surface facing upwards. A round probe was adapted to the testing machine and a 100 N load compressive force and 1.0 mm/min speed applied against the center of the bottom surface of the restoration to push out the filling. The force required for displacement or fracture was recorded in KgF and divided by the adhesive surface area (57 mm^2), calculated by the formula π (R + r)√(R-r)2 + H2), where 'R' is the radius of the top surface and 'r' is the radius of the bottom surface, converted into Mpa by the formula F / adhesion area.

Failure modes were analyzed under a dissecting microscope (Stereozoom, Bausch& Lomb, New York, EUA). Images were obtained in a 40x magnification and failure modes were classified as adhesive, cohesive, or mixed.

2.4 Nanoleakage

For nanoleakage analysis, three specimens from each group (n = 3) were immersed in 20 mL of distilled water and stored in an oven at 37ºC for 24 hours. The specimens were removed from the water and dried with absorbent paper and immersed in silver nitrate solution prepared with 25 g of silver nitrate crystals (Sigma Chemical Co., St. Louis, MO, USA) with pH = 11.0. Specimens were stored in dark sealed flasks for 24 hours, and after this period, they were washed with distilled water for 30 seconds and immersed in 20 mL of the photographic developer (Kodak Rochester, New York, USA) for 8 hours under fluorescent light. Then, the specimens were washed with distilled water for 30 seconds and polished underwater on a polishing machine (Metaserv 2000, Buehler, UK Ltd., Lake Bluff, IL, USA) using 600-, 1200- and 2000-grit sandpapers (Carbimet Disc Set, Buehler, UK Ltd., Lake Bluff, IL, USA - # 305208025) and 0.3 µ and 1 µ polishing pastes (Alpha Micro polish II Deagglomerateo Alumina, Buehler, UK Ltd., Lake Bluff, IL, USA) and felt disc (Buehler, UK Ltd., Lake Bluff, IL, USA). The specimens were demineralized by 37% phosphoric acid for 5 seconds, washed with distilled water for 30 seconds and dried with absorbent paper, and then left for 24 hours at room temperature (Chaves, et al., 2019). The specimens were mounted on a carbon-coated aluminum stand (Delton Vacuum Desk II, Moorestown, NJ) and the top and bottom interfaces were examined on a Scanning Electron Microscopy - SEM (Hitachi TM300) with 1000X magnification.

2.5 Statistical analysis

Normality of data was determined by a Kolmogorov-Smirnov test. Failure modes and nanoleakage were descriptively analyzed. Bond strength data were analyzed using two-way ANOVA and Tukey post-hoc test ($p<0.05$) using the IBM SPSS Statistics software for Windows (version 20.0; IBM Corporation, Armonk, NY, USA).

3. Results

3.1 Bond strength

Statistically significant interactions were found among chemical treatment versus aging time ($p<0,05$). Multiple comparisons among groups are shown in Table 2. In specimens aged for 24 hours, all chemical treatments provided similar values. In specimens aged for 6 months, the use of SBU alone or associated with silane provided higher values than the silane alone, however, it was not different from the group without chemical treatment. Aging for 6 months increased bond strength for all groups, except when silane alone was used.

Table 2: Means ± standard deviations of bond strength (MPa) according to chemical treatment, and aging time.

Chemical treatment	**24 horas**	**6 meses**
	Mean ± SD	**Mean ± SD**
GC	5,30±1,82Aa	8,80±3,07Ab
S	4,92±1,61Aa	6,52±1,67Ba
SBU	5,96±2,17Aa	8,83±3,03Ab
S+SBU	4,57±2,33Aa	9,79±3,27Ab

GC – Control group, S – Silane, SBU – Scotchbond universal, S+SBU – Silane+ Scotchbond universal.
Different capital letters indicate significant differences between surface chemical treatment for the same aging time (p<0.05). Different lowercase letters indicate significant differences among aging time for the surface chemical treatment (p<0.05).
Source: Personal archives, (2020).

3.2 Failure modes

The highest percentage of failures found after 24 hours was the adhesive mode. After 6 months, mixed failures were predominant when SBU and S+SBU were used as chemical treatments (Table 3).

Table 3: Failure mode distribution [(n (%)] according to chemical treatment, and aging time.

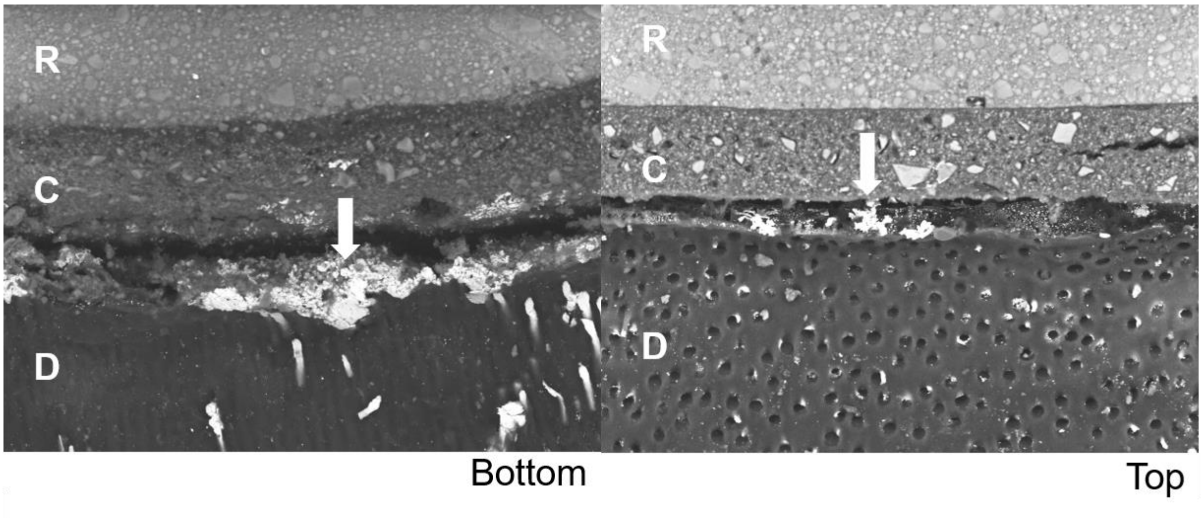
	Failure mode (24 hours)			**Failure mode (6 months)**		
Chemical Treatment	**Adhesive N (%)**	**Cohesive N (%)**	**Mixed N (%)**	**Adhesive N (%)**	**Cohesive N (%)**	**Mixed N (%)**
GC	**14(82.4)**	--	3(17.6)	**11(64.7)**	--	6(35.3)
S	**12(70.6)**	--	5(29.4)	**17(100)**	--	--
SBU	**12(70.6)**	1(5.9)	4(23.5)	4(23.5)	1(5.9)	**12(70.6)**
S+SBU	**13(76.5)**	1(5.9)	3(17.6)	8(47.1)	--	**9(52.9)**

GC – Control group, S – Silane, SBU – Scotchbond universal, S+SBU – Silane+ Scotchbond universal.
Source: Personal archives, (2020).

3.3 Nanoleakage

The qualitative measurement of silver crystals was done on the top and bottom surfaces of study groups in both aging times. A higher nanoleakage was found at the bottom surface compared with the top in both aging times, at the dentin-cement interface (Figure 2).

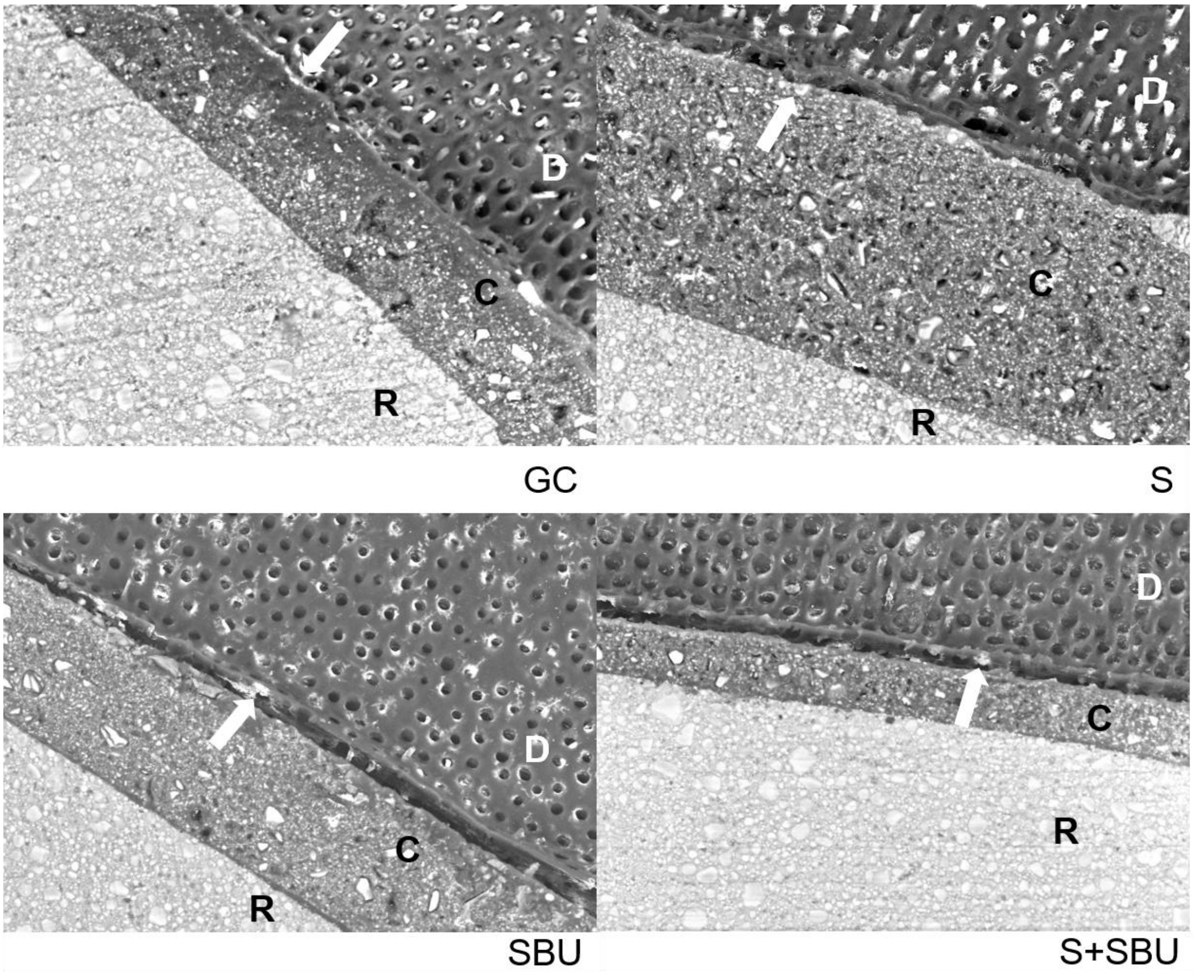
Figure 2: Photomicrograph (SEM) with 1000x magnification, showing the adhesive interface of the bottom and top of immediate samples.



(C) Cement, (D) Dentine, (R) Resin Composite, (Arrow) Silver crystal.
Source: Personal archives, (2020).

While at the cement-resin interface, no differences were found in the amount of silver crystals between the different chemical treatments applied to the external surface of the restoration (Figure 3).

Figure 3: Photomicrograph (SEM) with 1000x magnification, showing the adhesive interface of the top of aging samples.



(C) Cement, (D) Dentine, (R) Resin Composite, (Arrow) Silver crystal.
Source: Personal archives, (2020).

4. Discussion

The null hypothesis tested in this study was rejected for the bonding durability and the aging time, since the lower bonding durability was observed when silane was used alone as a chemical surface treatment after 6 months. However, the hypothesis was accepted for nanoleakage, as no differences were found in the amount of silver crystals at the cement-dentin interface between the chemical treatments applied and the aging time of the specimens.

ANOVA showed interaction between the study factors ($p<0,05$). The use of self-adhesive cement (RelyX U200) provided lower bond strength when used with silane as a chemical surface treatment. Silanes establish a covalent bond so that the polymerized resin matrix monomers form double carbon bonds and inorganic compounds by siloxane (Bähr, et al., 2013). RelyX U200 cement consists of methacrylate monomers modified with phosphoric acid that can demineralize dentin, and the cement infiltrates the hybrid layer with resin tags, without the need to previously remove the modified smear layer (Bulut, et al., 2018). Although, the main problem of the union of resinous compounds using silanes is the weakening of the bond (degradation) in the wet oral environment over time (Martinlinna, et al., 2018).

It is reported in the literature that the mechanical treatment with alumina blasting associated with the use of silane as a chemical treatment in indirect technique is used to reactivate the composite resin layer before cementation (Rodrigues, et al., 2009). However, it seems that this treatment is not necessary for the semi-direct technique since the composite resin is still chemically reactivated (Papacchini, et al., 2007b).

When SBU containing silane and 10MDP phosphate monomer was used in its composition, the durability of the bond was improved. This monomer has a high chemical affinity for calcium; is stable against hydrolysis and responsible for the interaction of cement with hydroxyapatite present in the dentin substrate (Turp, et al., 2013). Still, these results were not different from those found in the control group, where no chemical treatment was used, which can be explained by the presence of phosphoric acid and resinous monomers in the organic matrix of the self-adhesive cement, which demineralize the dentin superficially, remove the smear layer and provide chemical and micromechanical retention. The chemical bond is obtained through the bonding of resin monomers to calcium ions in hydroxyapatite (Santos, et al., 2011).

Aging also influenced the BS of semi-direct restorations, increasing its value except when S was used as chemical treatment. It is known that cements self-adhesive resins have their adhesion strength decreased in dentin, however, an increase in bond strength was observed in the study after 6 months of aging in water in the group in which no chemical treatment was used. The most accepted hypothesis is that the adhesion process involves groups of acid functional monomers that demineralize and infiltrate simultaneously the enamel and dentin, allowing adhesion through micromechanical retention and the chemical interaction between the monomer groups and the hydroxyapatite (Xuan & Wang, 2015). Bond strength also increased in groups that used only SBU or associated with Silane. This can be explained by a greater interaction promoted by the SBU from the start of dentin hybridization, whereas RelyX U200 requires a longer substrate maturation time and because this initial interaction is greater, its degradation would also be faster.

In the immediate analysis, the adhesive failure was the most common failure mode for RelyX U200. The adhesive failure occurred because the resistance of the adhesive interface was not greater than the resistance of the resin cement (Altinci, et al., 2018). This has also been observed in other studies and can indicate the limited ability of the cement to bond chemically and micro mechanically to dentin or the formation of a tooth-cement interface irregular, due to superficial demineralization by self-adhesive cement, compromising the formation of the hybrid layer (Lührs, et al., 2010; Rodrigues, et al., 2015). After 6 months of aging in distilled water, there was a change in the type of fracture from adhesive to mixed in the groups treated with SBU with or without silane, which is probably related to the decrease in the cohesive resistance of the resinous material and the adhesive interface simultaneously, compatible with greater hydrolytic degradation at the adhesive interface (Cura, et al., 2016).

A greater infiltration of silver crystals was found in the bottom of the restorations compared to the top surface in the cement-dentin interface for all groups in both storage times. There was minimal infiltration of silver crystals in the cement-resin interface that was not influenced by the chemical treatments applied. This adhesion process between cement and resin involves groups of monomers that simultaneously demineralize and infiltrate dentin, allowing greater adhesion through micromechanical retention and the chemical interaction between the monomer groups and the hydroxyapatite (Xuan & Wang, 2015).

The choice of the luting material and surface treatment can modify the interfacial characteristics of the composite and dentine, affecting the bond strength of cemented semi-direct restorations. Our study sheds new light on the issue and can guide dentists on their clinical practice in achieving higher success rates and better longevity of restorative treatments. Control of the environment and study variables is inherent to laboratory studies. Thus, other factors may influence the bond durability of semi-direct restoration when performed on the patient in a clinical environment, such as dental preparation, operator skill and restorative technique, which may represent limitations of this study. Clinical trials are recommended to confirm the laboratory results found in this study.

5. Conclusion

The study showed that silane as a chemical surface treatment associated with RelyX U200 cement obtained low performance in the bonding durability of semi-direct restorations. Chemical treatments did not influence the nano-infiltration of the cement-resin interface. The aging time influenced the bond strength and the failure mode of the restorations, but did not influence the nano-infiltration at the adhesive interface.

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