

**Effect of simplified and full-step surface treatments of indirect resin composite on dentin bonding stability and nanoleakage**

**Efeito de tratamentos de superfície simplificados e completos de resina composta indireta na estabilidade de união à dentina e nanoinfiltração**

**Efecto de tratamientos superficiales simplificados y completos de resina compuesta indirecta sobre la estabilidad de la unión dentinaria y la nanoinfiltración**

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**Joselúcia da Nóbrega Dias**

ORCID: <https://orcid.org/0000-0002-0435-2869>

Federal University of Rio Grande do Norte, Brazil

E-mail: [josynobrega14@gmail.com](mailto:josynobrega14@gmail.com)

**Andressa Eveline de Lima Ribeiro**

ORCID: <https://orcid.org/0000-0002-2371-1732>

Federal University of Rio Grande do Norte, Brazil

E-mail: [andressa.elima@hotmail.com](mailto:andressa.elima@hotmail.com)

**Ana Margarida dos Santos Melo**

ORCID: <https://orcid.org/0000-0001-7234-226X>

Federal University of Rio Grande do Norte, Brazil

E-mail: [anamdsmelo@gmail.com](mailto:anamdsmelo@gmail.com)

**Boniek Castillo Dutra Borges**

ORCID: <https://orcid.org/0000-0003-4313-5776>

Federal University of Rio Grande do Norte, Brazil

E-mail: [boniek.castillo@gmail.com](mailto:boniek.castillo@gmail.com)

**Isauremi Vieira de Assunção**

ORCID: <https://orcid.org/0000-0003-3171-6514>

Federal University of Rio Grande do Norte, Brazil

E-mail: [isauremi@gmail.com](mailto:isauremi@gmail.com)

**Abstract**

The study evaluated the effect of the application of simplified or full-step surface treatments and aging times on dentin bonding stability and nanoleakage of indirect resin composite

restorations. This is an in vitro study with one hundred and sixty indirect resin composite restorations (4.8 x 2.8 x 4.0 mm) produced to fill dentin preparations in bovine dentin. The specimens were divided into eight groups according to chemical treatment [No treatment; Silane; Scotchbond™ Universal adhesive (SBU); and Silane + SBU], and aging time in water [24 h and six months]. Push-out bond strength (PBS) was evaluated by 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 posthoc tests ( $p < 0.05$ ) were used to compare PBS among the groups, while failure modes and nanoleakage were analyzed descriptively. The bonding stability of indirect resin composite restorations was influenced by chemical surface treatments and aging times ( $p < 0.05$ ). Significant differences were determined between chemical treatment applied, mainly when SBU was used after aging the samples. The different surface treatments and aging time did not influence the nanoleakage at the cement-resin interface. The study results suggest that the simplified surface treatment using a universal adhesive improved the dentin bonding stability of indirect resin composite restorations.

**Keywords:** Adhesiveness; Resin cements; Dental restoration failure; Scanning electron microscopy.

### Resumo

O estudo avaliou o efeito da aplicação de tratamentos de superfície simplificados ou completos e os tempos de envelhecimento sobre a estabilidade da união à dentina e nanoinfiltração de restaurações de resina composta indireta. Trata-se de um estudo in vitro com cento e sessenta restaurações indiretas de resina composta (4,8 x 2,8 x 4,0 mm) produzidas em preparos confeccionados em dentes bovinos. Os espécimes foram divididos em oito grupos de acordo com o tratamento químico [Sem tratamento; Silano; Adesivo Single Bond Universal™ (SBU); e Silano + SBU], e tempo de envelhecimento em água [24 horas e seis meses]. A resistência de união por push-out (RU) foi avaliada por 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 comparar a RU entre os grupos, enquanto os modos de falha e nanoinfiltração foram analisados descritivamente. A estabilidade de união de restaurações indiretas de resina composta foi influenciada por tratamentos químicos de superfície e tempos de envelhecimento ( $p < 0,05$ ). Diferenças significativas foram determinadas entre os tratamentos químicos aplicados, principalmente quando o SBU foi

utilizado após o envelhecimento das amostras. Os diferentes tratamentos de superfície e tempo de envelhecimento não influenciaram a nanoinfiltração na interface cimento-resina. Os resultados do estudo sugerem que o tratamento simplificado da superfície com adesivo universal melhorou a estabilidade da união dentinária de restaurações indiretas de resina composta.

**Palavras-chave:** Adesividade; Cimentos de resina; Falha de restauração dentária; Microscopia eletrônica de varredura.

### **Resumen**

El estudio evaluó el efecto de la aplicación de tratamientos superficiales simplificados o completos y los tiempos de envejecimiento sobre la estabilidad de la unión de dentina y la nanoinfiltración de restauraciones indirectas de resina compuesta. Este es un estudio in vitro con ciento sesenta restauraciones indirectas de resina compuesta (4,8 x 2,8 x 4,0 mm) para rellenar preparaciones de dentina en dentina bovina. Las muestras se dividieron en ocho grupos según el tratamiento químico [Sin tratamiento; Silano; Single Bond Universal™ (SBU); y Silano + SBU], y tiempo de crianza en agua [24 hy seis meses]. La resistencia de unión por expulsión (RU) se evaluó mediante una máquina de prueba universal (1,0 mm / min), los modos de falla mediante un microscopio de disección y la nanoinfiltración mediante microscopía electrónica de barrido. Se utilizaron ANOVA dos factores y post hoc de Tukey ( $p < 0.05$ ) para comparar RU entre grupos, mientras que los modos de falla y nanoinfiltración se analizaron descriptivamente. La estabilidad de la unión de las restauraciones indirectas de resina compuesta se vio influenciada por los tratamientos químicos de la superficie y los tiempos de envejecimiento ( $p < 0,05$ ). Se determinaron diferencias significativas entre los tratamientos químicos aplicados, principalmente cuando se utilizó el SBU después del envejecimiento de las muestras. Los diferentes tratamientos superficiales y el tiempo de envejecimiento no influyeron en la nanoinfiltración en la interfaz cemento-resina. Los resultados del estudio sugieren que el tratamiento simplificado de la superficie con adhesivo universal mejoró la estabilidad de la unión dentinaria de las restauraciones indirectas de resina compuesta.

**Palabras clave:** Adhesividad; Cementos de resina; Fallo de la restauración dental; Microscopía electrónica de barrido.

## 1. Introduction

Indirect resin composite restorations (IRCR) can be a viable alternative when there is a considerable loss of tooth structure in posterior teeth that require onlays, inlays or overlays restorations, non-cariou cervical lesions, and class II preparations (Caneppele, et al., 2020; Torres, et al., 2020). IRCR does not require the restoration's laboratory processing since the proper professional can produce them in the office, saving time and reducing operational costs (Alharbi, et al., 2018). However, durable bonding between IRCR and luting agents is detrimental for long-lasting clinical success, which depends on a reliable interaction on substrate/luting agent and luting agent/dentin interfaces (Matinlinna, et al., 2018).

Adhesion to dentin remains a significant challenge for restorative dentistry. It represents one of the points for cemented restorations' clinical success (Sokolowsky, et al., 2018). Thus, the bonding stability and the risk of nanoleakage depend on the cement-dentin bonding and can be influenced by several factors, including the surface treatment of IRCR (Matinlinna, et al., 2018). To improve the interaction on the interface between indirect resin composite substrate and resin-based luting agent, sandblasting with alumina oxide, application of silane, and universal adhesive systems on the indirect substrate have been suggested, which increases dentin bond strength (Campos, et al., 2020). In this way, the results of a systematic review showed that using scotchbond universal adhesives without previous substrate silanization can provide adequate bond strength of luting agents to indirect resin composite substrates (Cuevas-Suárez, et al., 2020), which would simplify bonding steps and save chair time. Considering IRCR, the substrate sandblasting would require an additional laboratory step, impairing the professional to perform a fast in-office procedure (Silva Júnior, et al., 2017). In this way, only chemical treatments of the indirect substrate with silane associated or not to an adhesive system have been performed before bonding IRCR (Isolan, et al., 2014).

Self-etching dual resin cements are associated with self-etching/universal adhesive systems specific to the cement itself. This system promotes the preparation surface treatment before cementation, favors hybridization, and better retains the restoration (Miotti, et al., 2020). Another advantage of these cements is the chemical interaction with their respective adhesive system, which allows the simultaneous polymerization of these components during cementation, promoting a complete polymerization of the resin cement, improved retention, and fracture resistance of the restoration (Itthipongstorn, et al., 2020).

On the other hand, the effect of universal adhesive with or without previous silanization of IRCR is still unclear, especially on dentin bonding stability and nanoleakage. Studies indicate that universal adhesives modify the interfacial characteristics of a wide range of substrates. However, little is known about the effectiveness of using silane alone or silane-containing adhesive in the surface treatment of IRCR, as well as about the longevity of bond of cement RelyX Ultimate + Scotchbond Universal (SBU) in this type of restoration.

This *in vitro* study aimed to evaluate the effect of silane application in conjunction or not with a universal adhesive system and aging times on dentin bonding stability and nanoleakage of IRCR. The null hypothesis was that the dentin bonding stability and nanoleakage of IRCR would not be influenced by the tested chemical treatments and aging times.

## **2. Material and methods**

### **2.1 Experimental design**

This *in vitro* study was carried out at the Dentistry Department of the Federal University of Rio Grande do Norte. The response variables were: Bond Strength (BS), Failure Mode (FM), and Nanoleakage (NL). For BS and FM, a 4 x 2 factorial design was involved: four chemical treatments [no treatment, Silane, Scotchbond™ Universal adhesive (SBU), and Silane (S) + SBU]; and two water aging times [24 h (non-aged), and six months]. For NL, a 4 x 2 x 2 was involved: four chemical treatments, two water aging times, and two adhesive interfaces (top and bottom). The materials used in this study are described in Table 1.

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

Product	Manufacturer	Composition <sup>a</sup>	Lot
<b>Filtek Z250XT A2</b>	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
<b>RelyX Ultimate A2</b>	3M ESPE, St. Paul, MN, USA	Base paste: silane-treated glass powder; 2-propenoic acid; 2-methyl-, 1,10-[1-(hydroxymethyl)-1,2-ethanediyl], ester reaction products with 2-hydroxy-1,3-propanediyl, dimethacrylate and phosphorous oxide; TEGDMA; silane-treated silica; oxide glass chemicals; sodium persulfate; tert-butyl peroxy-3,5,5-trimethylhexanoate; and copper (II) acetate monohydrate. Catalyst paste: silane-treated glass powder; dimethacrylate; silane-treated silica; 1-benzyl-5-phenylbarbic-acid; calcium salt; sodium p-toluenesulfinate; 1,12-dodecane dimethacrylate; calcium hydroxide; 2-propenoic acid, 2-methyl-, [3-methoxypropyl]imino]di-2,1-thanediyl ester; and titanium dioxide.	1803700434
<b>Scotchbond Universal</b>	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
<b>RelyX Ceramic Primer</b>	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.*

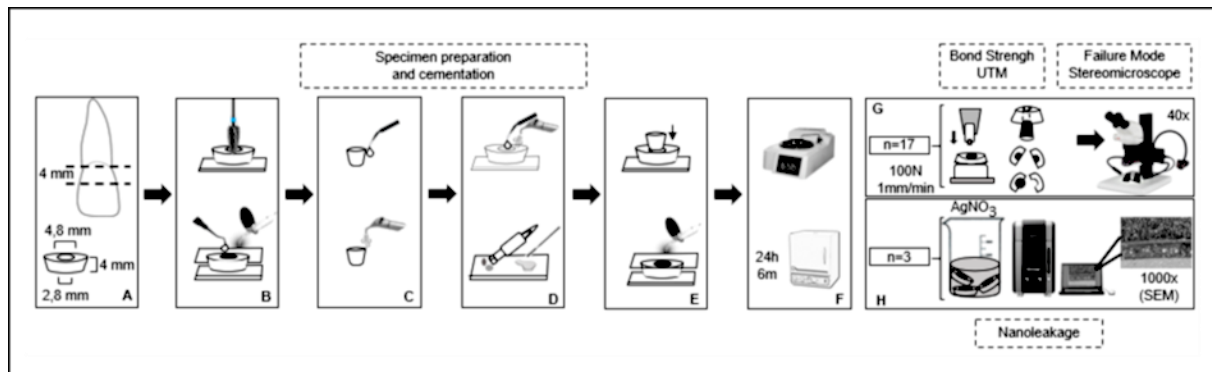
<sup>a</sup> Data supplied by the manufacturer.

Source: Authors, (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 °C for one week. The roots were removed by cutting at the cemento-enamel 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. A 4.0-mm-thick disc with the pulp cavity in the center was obtained. The specimens' top and bottom surfaces 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 ten preparations (Souza-Lima, et al., 2020). 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 seconds (Coltolux® LED, Coltène, Altstätten, Switzerland – 1264 mW/cm<sup>2</sup>). The fillings were removed from the preparations and heat-cured in a microwave oven (Consul, SC, Brazil), for 5 minutes at power 800 W<sup>2</sup> (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 restoration surface with a flexible micro-brush (Kg Brush, KG Sorensen, São Paulo, Brazil), the SBU was actively applied for 20 seconds, while silane was applied for 60 seconds. Soon after, the restoration was cemented in the preparation. An SBU layer was actively applied for 20 seconds to the preparation and gently air-dried for 5 seconds. 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 cement excess was removed with a micro-brush, and light-cured for 20 seconds. Then, half of the specimens were stored 24 hours and a half for six months in distilled water changed weekly, in a drying oven (SL-100, Solab, Piracicaba, São Paulo, Brazil) at 37°C, before bond strength, failure mode, and nanoleakage analyses. All procedures were performed by two previously calibrated researchers. The study protocol is shown in Figure 1.

**Figure 1.** Schematic representation of the methods.



*Bovine incisors were cut (A); Standardized tooth preparations (4.8x2.8x4mm) were created and restored (B); Chemical treatments were applied to the surface of the restoration (C); Cavities received SBU (D) and Restorations were cemented (E); Restorations were polished and stored for 24 hours and 6 months in water (F); The specimens (n=17) were submitted to the push-out bond strength test in a universal testing machine and failure modes were analyzed under a dissecting microscope (G); Three samples from each group (n=3) were submitted to nanoleakage analysis in Scanning Electron Microscopy (H).*

Source: Authors, (2020).

### 2.3 Push-out bond strength and failure modes

The specimens (n = 17) were placed with the bottom surface facing upwards. A round probe was adapted to the universal testing machine (Microtensile OM150, Odeme, Luzerna, SC, Brazil). A 100 N load compressive force applied against the center of the restoration's bottom surface to push out the filling (1.0 mm/min). The force required to debond was recorded in N and converted to MPa.

### 2.4 Failure modes

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. In cohesive failure, failure is observed only in dentin or only in the resin composite. Adhesive failure occurs at the cement-dentin adhesive interface, without fracturing the dental element. And in mixed failure, the fracture occurs simultaneously in the dentin and /or resinous material and at the adhesive interface.



## 2.5 Nanoleakage

Three additional specimens from each group ( $n = 3$ ) were produced, immersed in 20 mL of distilled water, and stored in a drying oven (SL-100, Solab, Piracicaba, São Paulo, Brazil) at 37°C, for 24 hours. The specimens were removed from the water, 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. After this period, they were washed with distilled water for 30 seconds and immersed in 20 mL of a photographic developer (Kodak Rochester, New York, USA) for eight hours under fluorescent light. 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). Polishing pastes - 0.3  $\mu$  and 1  $\mu$  (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, dried with absorbent paper, and then left 24 hours at room temperature (Chaves, et al., 2019). The specimens were mounted on carbon-coated aluminum support (Delton Vacuum Desk II, Moorestown, NJ), and four quadrants of the top and bottom interfaces were examined on a Scanning Electron Microscopy - SEM (Hitachi TM300) with 2000X magnification.

## 2.6 Statistical analysis

Failure modes and nanoleakage were descriptively analyzed. Bond strength data were analyzed using two-way ANOVA (Table 2) and Tukey posthoc test ( $p < 0.05$ ) using the IBM SPSS Statistics software for Windows (version 20.0; IBM Corporation, Armonk, NY, USA).

**Table 2.** Results of two-way ANOVA test.

Source of Variation	Sum of squares	d.f.	Mean square	F value	P value
Surface treatment	243,7	3	81,25	11,41	<0,0001
Aging time	261,6	1	261,6	36,75	<0,0001
Interaction	56,42	3	18,81	2,642	0,0521
Residual	911,1	128	7,118		

\*Significantly different at  $P < 0,05$ . Source: Authors, (2020).

### 3. Results

#### 3.1 Bond strength

Two-way ANOVA (Table 3) indicated that the bond strength was significantly influenced individually by surface treatment and aging time ( $p < 0,05$ ). Multiple comparisons among the groups are shown in Table 3. In non-aged specimens (24 h), C, S, and SBU groups showed statistically similar bond strength values, which were higher than S + SBU group. However, SBU provided statistically higher values than other chemical treatments, which were similar. Aging for six months decreased statistically bond strength values in C and S groups compared to 24 hours and maintained in SBU and S+SBU groups.

**Table 3.** Means  $\pm$  standard deviations of bond strength (MPa) according to the chemical treatment and aging time tested.

Chemical treatment	Aging	
	24 hours (non-aged)	6 months
C	15,08 $\pm$ 2,64 Aa	10,65 $\pm$ 2,97 Bb
S	14,94 $\pm$ 3,10 Aa	11,31 $\pm$ 2,23 Bb
SBU	15,71 $\pm$ 3,39 Aa	14,27 $\pm$ 0,99 Aa
S+SBU	12,01 $\pm$ 3,32 Ba	10,42 $\pm$ 1,74 Ba

C – 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 surface chemical treatment ( $p < 0.05$ ). Source: Authors, (2020).

### 3.2 Failure modes

The highest percentage of failures found after 24 hours was the mixed-mode in all groups. After six months, mixed failures were predominant when SBU and S+SBU were used as chemical treatments (Table 4).

**Table 4.** Failure mode distribution [(n (%))] according to chemical treatment and aging time tested.

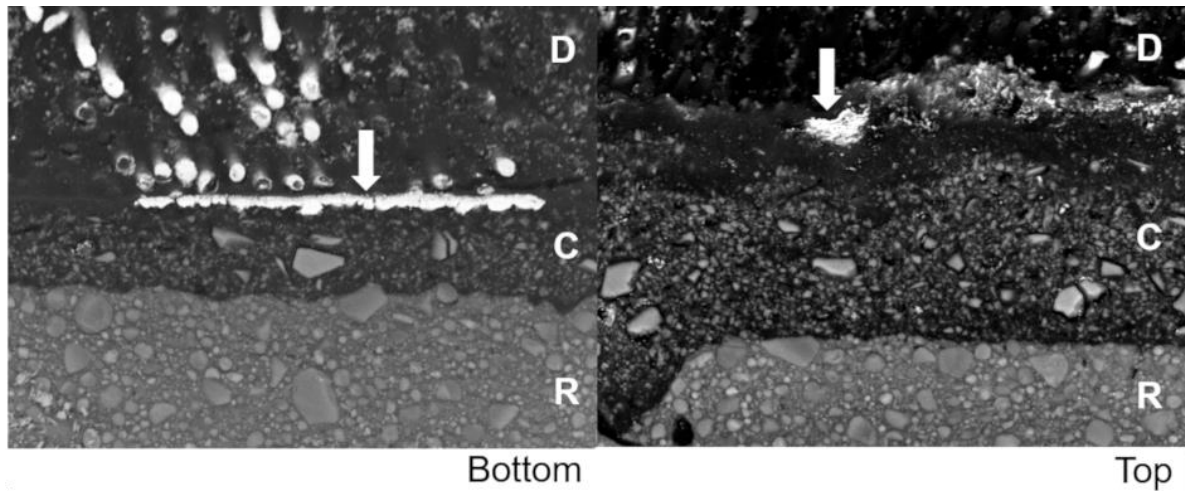
Chemical treatment	Aging					
	24 hours (non-aged)			6 months		
	Adhesive	Cohesive	Mixed	Adhesive	Cohesive	Mixed
C	--	4 (23.5)	13 (76.5)	--	10 (58.8)	7 (41.2)
S	--	5 (29.4)	12 (70.6)	1(5.9)	10 (58.8)	6 (35.3)
SBU	--	1 (5.9)	16 (94.1)	--	3 (17.6)	14 (82.4)
S+SBU	--	1 (5.9)	16 (94.1)	--	8 (47.1)	9 (52.9)

C – Control group, S – Silane, SBU – Scotchbond universal, S + SBU – Silane + Scotchbond universal. Source: Authors, (2020).

### 3.3 Nanoleakage

The qualitative analysis of silver crystals uptake was performed on the top and bottom surfaces of non-aged and aged specimens. In general, higher nanoleakage was found at the bottom surface than in the top (Figure 2) for all groups at the cement-dentin interface. In the immediate analysis (Figure 3) and the aged analysis (Figure 4), the surface treatments applied in the restoration did not influence the nanoleakage at the cement-resin interface. At the cement-dentin interface, minimal nanoleakage occurred with a similar pattern among the groups.

**Figure 2.** Photomicrograph (SEM) with 2000x magnification, showing the adhesive interface of the bottom and top of immediate samples without chemical.

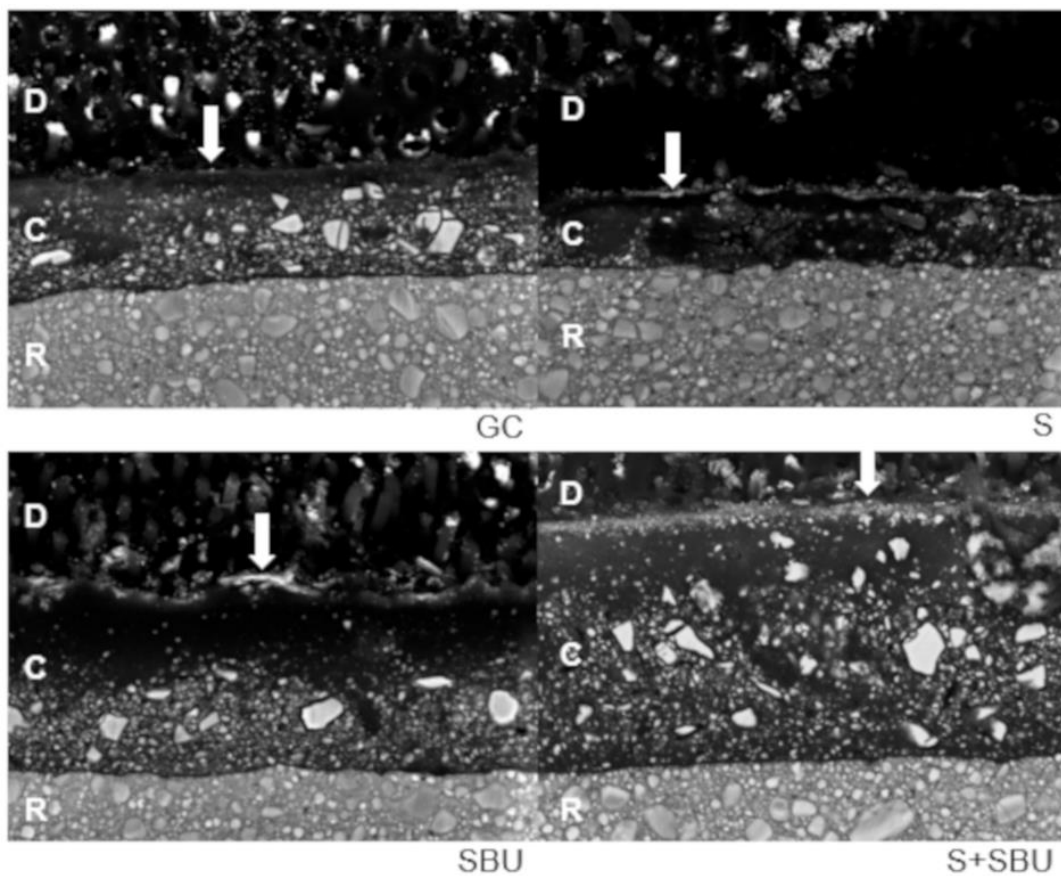


(R) Resin, (D) Dentine, (C) Cement, (Arrow) Silver crystal.

Source: Authors, (2020).

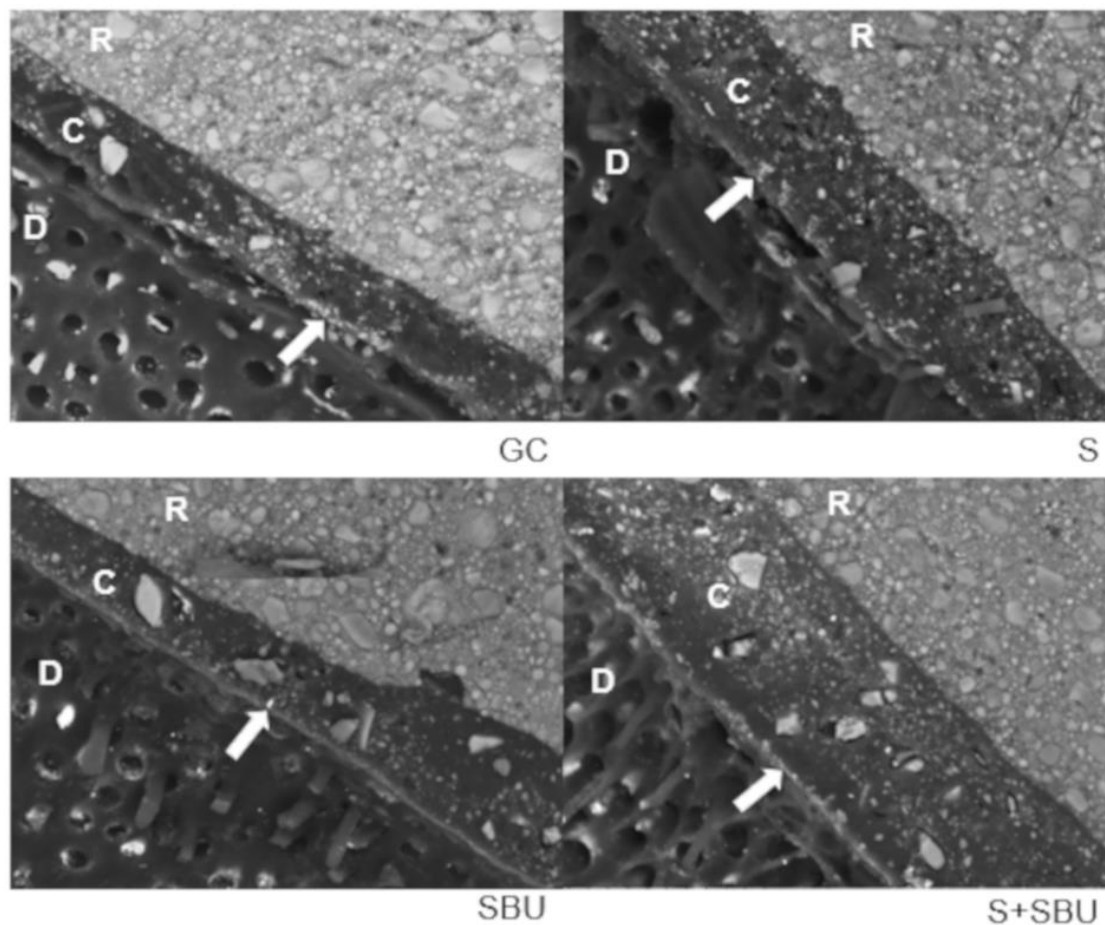
**Figure 3.** Photomicrograph (SEM) with 2000x magnification, showing the adhesive interface of the top of immediate samples with different surface treatments.

(R)



Resin, (D) Dentine, (C) Cement, (Arrow) Silver crystal. Source: Authors, (2020).

**Figure 4.** Photomicrograph (SEM) with 2000x magnification, showing the adhesive interface of the top of aged samples with different surface treatments.



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

#### 4. Discussion

The null hypothesis tested in this study – that dentin bonding stability and nanoleakage of IRCR would not be influenced by the tested chemical treatments and aging times - was rejected.

Higher bond strength values were found when SBU was used as a chemical treatment after six months. Possibly, the improved wettability of the adhesive associated with its strong chemical interaction with the substrate provided by 10-MDP favors higher values of BS in aged and non-aged samples, which was also found by Tsujimoto, et al. (2017). Therefore, the clinical procedure for cementing the IRCR can be simplified and more efficient with the use of Scotchbond Universal adhesive, which contains silane and 10-MDP, as indicated by the systematic review developed by Cuevas-Suárez, et al. (2020). Besides, universal adhesives

can promote hybridization in dentin through superficial demineralization, facilitating the infiltration and polymerization of resin cement to create a micromechanical retention (Miotti, et al., 2020).

On the other hand, a harmful interference in silane use is suggested, mainly after six months of aging. The silane is composed of a bifunctional molecule, which establishes chemical bonds with organic molecules of the resin composite and inorganic components of the resin cement through one of its functional groups. Possibly, the lowest BS of the groups that used S alone or associated with the SBU occurred due to a weak chemical interaction with the composite resin or the resin cement. Perhaps the low amount of inorganic molecules available in the self-etching resin cement has impaired this interaction with silane and, consequently, reduced the bond between the resin cement and the surface of the composite resin restoration (Oliveira, et al., 2019). These results corroborate with other studies whose authors stated that silane alone could not establish all the necessary chemical bonds to increase the BS at the adhesive interface (Yoshihara, et al., 2016).

In addition to chemical surface treatment, aging also influenced the BS of IRCR. The fact that specimens treated with SBU showed better BS results could be explained by a stronger interaction promoted by the SBU associated with self-etching cement RelyX Ultimate from the start of dentin hybridization. The adhesive hydrophilicity is suggested as the reason for the degradation of the adhesive interface by water absorption. However, incorporating a hydrophobic monomer, 10 MDP, retains its hydrolytic stability, which may explain the maintenance of the BS of the restorations treated with SBU after six months (Feitosa, et al., 2014).

The results of a recent study showed that the use of a resin cement associated with an adhesive system was able to promote greater bond strength to dentin, both immediately and in the long term (Miotti, et al., 2020). Also, RelyX Ultimate resin cement incorporates a dark polymerization activator from the SBU catalyst paste, which may have contributed to its better performance mainly when the adhesive is not cured, as in the present study, this would be especially true after aging because this double cure has been described as slower (Yoshida & Atsuta, 2006). On the other hand, when using self-adhesive resin cement for luting semi-direct restorations in composite resin, there is no difference when using SBU associated or not with silane (Dias, et al., 2020).

After the bond strength test, the samples were analyzed for the type of fracture in a stereomicroscope. It was possible to observe that the mixed failure was predominant after 24 hours of storage, while after six months, the mixed failure mode was the most common when

SBU and S + SBU were used as chemical surface treatments. These results probably occurred due to the higher BS to the dentin promoted by the SBU penetration in the smear layer, positively interfering in the hybrid layer strength (Yoshihara, et al., 2013). Also, resin composites containing UDMA, such as Filtek Z250, tend to absorb more water, causing hydrolysis. This hydrolysis impairs the chemical bond between the filler particles and the matrix, promoting detachment of the hydrolyzed particles from the matrix, which reduces the material strength (Fonseca, et al., 2017; Altinci, et al., 2018).

More significant infiltration of silver crystals at the dentin-cement interface was found in the bottom of the restorations than in the top surface, regardless of the chemical surface treatment. It is possible that poorer curing of the materials occurred in the bottom due to less light penetration, consequently affecting adhesion. This phenomenon can induce the early formation of cracks in the tooth-restoration interface, leading to postoperative sensitivity, discoloration, and bacterial infiltration with consequent reduction at clinical longevity (Ferracane & Hilton, 2016).

Laboratory studies are performed in controlled environments and, therefore, have limitations as they eliminate clinical factors that can influence the longevity of indirect adhesive restorations, such as the form of preparation, the operator's experience, as well as the type of material and restorative technique used. In this way, clinical trials are necessary to confirm the laboratory results found in this study.

## **5. Conclusion**

This study showed that the use of simplified chemical treatment with SBU adhesive, without silane, improved the bonding stability over time of the IRCR cemented with self-etching resin cement. Nanoleakage at the cement-resin interface was not influenced by the type of surface treatment or by the aging time of the restorations.

## **References**

Alharbi, A., Rocca, G. T., Dietschi, D., & Krejci, I. (2014). Semidirect composite onlay with cavity sealing: a review of clinical procedures. *Journal of esthetic and restorative dentistry*, 26(2), 97-106.

Altinci, P., Mutluay, M., & Tezvergil-Mutluay, A. (2017). Repair bond strength of nanohybrid composite resins with a universal adhesive. *Acta biomaterialia odontologica Scandinavica*, 4(1), 10–19.

Campos, M., Moura, D., Borges, B., Assuncao, I. V., Caldas, M., Platt, J. A., Özcan, M., & Souza, R. (2020). Influence of Acid Etching and Universal Adhesives on the Bond Strength to Dentin. *Brazilian dental journal*, 31(3), 272–280.

Caneppele, T., Meirelles, L., Rocha, R. S., Gonçalves, L. L., Ávila, D., Gonçalves, S., & Bresciani, E. (2020). A 2-year clinical evaluation of direct and semi-direct resin composite restorations in non-carious cervical lesions: a randomized clinical study. *Clinical oral investigations*, 24(3), 1321–1331.

Chaves, L., Oliveira, S. N., Özcan, M., Acchar, W., Caldas, M., Assunção, I. V., Souza, R., & Borges, B. (2019). Interfacial Properties and Bottom/Top Hardness Ratio Produced by Bulk Fill Composites in Dentin Cavities. *Brazilian dental journal*, 30(5), 476–483.

Cuevas-Suárez, C. E., de Oliveira da Rosa, W. L., Vitti, R. P., da Silva, A. F., & Piva, E. (2020). Bonding Strength of Universal Adhesives to Indirect Substrates: A Meta-Analysis of in Vitro Studies. *Journal of prosthodontics*, 29(4), 298–308.

Dias, J. N., Ribeiro, A. E. L., Melo, A. M. S., Borges, B. C. D., & Assunção, I. (2020). Impact of simplified chemical treatment of semi-direct restorations cemented with self-adhesive resin cement on dentin bond durability and nanoleakage. *Research, Society and Development*, 9 (11), 1-18.

Feitosa, V. P., Sauro, S., Ogliari, F. A., Ogliari, A. O., Yoshihara, K., Zanchi, C. H., Correr-Sobrinho, L., Sinhorette, M. A., Correr, A. B., Watson, T. F., & Van Meerbeek, B. (2014). Impact of hydrophilicity and length of spacer chains on the bonding of functional monomers. *Dental materials*, 30(12), 317–323.

Ferracane, J. L., & Hilton, T. J. (2016). Polymerization stress--is it clinically meaningful?. *Dental materials*, 32(1), 1–10.



Fonseca, A. S., Labruna Moreira, A. D., de Albuquerque, P. P., de Menezes, L. R., Pfeifer, C. S., & Schneider, L. F. (2017). Effect of monomer type on the CC degree of conversion, water sorption and solubility, and color stability of model dental composites. *Dental materials*, 33(4), 394–401.

Godoy, C. E., Brum, R. T., & Higashi, C. (2014). Semi-direct technique for partial reconstructions in posterior teeth - case report. *Full dentistry in science*, 5(17), 124-133.

Isolan, C. P., Valente, L. L., Münchow, E. A., Basso, G. R., Pimentel, A. H., Schwantz, J. K., Silva, A. V., & Moraes, R. R. (2014). Bond strength of a universal bonding agent and other contemporary dental adhesives applied on enamel, dentin, composite, and porcelain. *Applied Adhesion Science*, 2(25), 1-10.

Itthipongsatorn, N., & Srisawasdi, S. (2020). Dentin microshear bond strength of various resin luting agents to zirconia-reinforced lithium silicate ceramics. *The Journal of prosthetic dentistry*, 124(2), 231–237.

Matinlinna, J. P., Lung, C., & Tsoi, J. (2018). Silane adhesion mechanism in dental applications and surface treatments: A review. *Dental materials*, 34(1), 13–28.

Miotti, L. L., Follak, A. C., Montagner, A. F., Pozzobon, R. T., da Silveira, B. L., & Susin, A. H. (2020). Is Conventional Resin Cement Adhesive Performance to Dentin Better Than Self-adhesive? A Systematic Review and Meta-Analysis of Laboratory Studies. *Operative dentistry*, 45(5), 484–495.

Oliveira, P., Oliveira, L., Cabral, M. R., De Vito Moraes, A. G., Sgura, R., Cesar, P. F., Gonçalves, M., Brugnera Junior, A., & Bussadori, S. K. (2019). Effect of Surface Treatment with CO2 Laser on Bond Strength in Composite Resin Restorations. *Photobiomodulation, photomedicine, and laser surgery*, 37(7), 428–433.

Silva Júnior, M. E., Ohata, G., Tonetto, M. R., Campos, E. A., & Andrade, M. F. (2017). Adhesive Restoration in Posterior Teeth using Semidirect Technique. *World journal of dentistry*, 8(5), 413-416.

Sokolowski, G., Szczesio, A., Bociong, K., Kaluzinska, K., Lapinska, B., Sokolowski, J., Domarecka, M., & Lukomska-Szymanska, M. (2018). Dental Resin Cements-The Influence of Water Sorption on Contraction Stress Changes and Hygroscopic Expansion. *Materials (Basel, Switzerland)*, 11(6), 973.

Sousa-Lima, R. X., Melo, A. M. S., Guimarães, L. K. C., Souza, R. O. A., Caldas, M. R. G. R., Assunção, I. V., & Borges, B. C. D. (2020). Mechanical properties of low and regular viscosity bulk fill composites in a 3D dentin cavity model. *Journal of adhesion science and technology*, 35.

Torres, C., Mailart, M. C., Crastechini, É., Feitosa, F. A., Esteves, S., Di Nicoló, R., & Borges, A. B. (2020). A randomized clinical trial of class II composite restorations using direct and semidirect techniques. *Clinical oral investigations*, 24(2), 1053–1063.

Tsujimoto, A., Barkmeier, W. W., Takamizawa, T., Wilwerding, T. M., Latta, M. A., & Miyazaki, M. (2017). Interfacial Characteristics and Bond Durability of Universal Adhesive to Various Substrates. *Operative dentistry*, 42(2), 59–70.

Yoshida, K., & Atsuta, M. (2006). Post-irradiation hardening of dual-cured and light-cured resin cements through machinable ceramics. *American journal of dentistry*, 19(5), 303–307.

Yoshihara, K., Nagaoka, N., Sonoda, A., Maruo, Y., Makita, Y., Okihara, T., Irie, M., Yoshida, Y., & Van Meerbeek, B. (2016). Effectiveness and stability of silane coupling agent incorporated in 'universal' adhesives. *Dental materials*, 32(10), 1218–1225.

Yoshihara, K., Yoshida, Y., Nagaoka, N., Hayakawa, S., Okihara, T., De Munck, J., Maruo, Y., Nishigawa, G., Minagi, S., Osaka, A., & Van Meerbeek, B. (2013). Adhesive interfacial interaction affected by different carbon-chain monomers. *Dental materials*, 29(8), 888–897.

**Percentage of contribution of each author in the manuscript**

Joselúcia da Nóbrega Dias – 20%

Andressa Eveline de Lima Ribeiro – 20%

Ana Margarida dos Santos Melo – 20%

Boniek Castillo Dutra Borges – 20%

Isauremi Vieira de Assunção – 20%