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Shrinkage stress, long-term adaptation and bond strength of low-shrinkage composite resins

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ABSTRACT

Purpose: To evaluate the internal adaptation, bond strength, and polymerization stress of silorane- and methacrylate-based composite resins.

Material and methods: Three methacrylate-based composite resins (Heliomolar; Tetric N-Ceram and Aelite LS) and one silorane-based composite resin (Filtek Silorane) were tested. Polymerization stress $(n=5)$ was determined by the insertion of the composite resin between rods of polymethyl methacrylate. The ratio of the maximum force of contraction was recorded and the cross-sectional area of the rod was used to calculate the nominal stress. Bond strength was evaluated by microtensile bond test. Dentin surfaces of human third molars were bonded, sectioned, and stored for 24 h or 1 year in distilled water before the bond strength test. The ratio of maximum force and the adhered area was used for the bond strength calculation. For internal adaptation analysis, third molars received Class II cavities and were restored according to either an incremental oblique or bulk-filling technique. After being sectioned perpendicularly, impressions were taken and epoxy resin replicas were obtained of the internal surfaces of the restorations (after 24 h and 1 year of storage) to analyze gap formation using scanning electron microscopy.

Results: Filtek Silorane showed the highest bond strength after one year of storage, the lowest formation of gaps, and polymerization stress similar to methacrylate-based materials.

Conclusion: Silorane restorative material presented polymerization stress comparable to that of methacrylate-based composite resins, stable dentin bond strength after one year and better internal adaptation to the cavity walls, showing good alternative to traditional composite resins and promising longevity.

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1. Introduction

The basic composition of composite resins involves monomers (such as bis-phenol A diglycidyl methacrylate, urethane dimethacrylate and triethylene glycol dimethacrylate), fillers, inhibitors, silane couple agent and photoinitiators $[1,2]$. The polymerization reaction of composite resins produces volumetric shrinkage of approximately 3– 5%, which causes tooth structural damages and problems at bonded interface, depending on the type of cavity preparation, volume of material polymerized, and how this composite resin was placed [\[3](#page-9-0)–[5\].](#page-9-0)

In an attempt to reduce or overcome the effects of polymerization shrinkage, techniques for incremental placement [\[6,7\],](#page-9-0) different lightcuring sources, irradiation techniques [\[8\],](#page-9-0) and use of low-shrinkage

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composite resins or a low-modulus intermediate flowable layer, known as the elastic wall concept $[9]$, have been proposed. Lowshrinkage composite resins present new monomeric formulation or higher filler loading that decreases organic content of commercial composite resins [\[1,2,5\].](#page-9-0) Formulations of new composite resins require time and expertize in the field of polymers; additions and composition changes must not compromise the physical properties and handling of composite resin.

A specific posterior composite resin was developed based on monomers siloxane and oxirane (Filtek Silorane, 3M ESPE, St. Paul, MN, USA) instead of traditional methacrylates. The polymerization reaction for these compounds is different from that of methacrylates and involves the opening of the oxirane ring [\[10\].](#page-9-0) Because resin matrix of silorane composite resin significantly differs from that of conventional methacrylate-based composite resins, a new bonding agent needed to be used with silorane composite resin. Filtek Silorane is therefore presented with two-step self-etch primer, called Silorane System Adhesive (3M ESPE). This adhesive has features of

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conventional methacrylate adhesives, especially in regard to its bonding mechanism to dentin. An adaptation was needed to make it compatible with the highly hydrophobic silorane matrix [\[11\]](#page-9-0). Aelite LS (Bisco, Inc., Schaumburg, IL USA) presents high amount of filler particles with glass and amorphous silica (84–88% by weight and 74–76% by volume) to reduce the polymerization shrinkage (1.4–1.9% volumetric shrinkage and 0.5–0.6% linear shrinkage) according to its manufacturer.

Although polymerization shrinkage stress reduction is clinically desirable, especially with posterior teeth composite resins [\[12\],](#page-9-0) little is known relative to these materials, such as shape of convenience, bond strength of composite resins using specific bonding agents, and the adaptation of the restorative material in internal cavity walls using Class II cavities. The aim of this study was to evaluate the polymerization stress, the internal adaptation on cavities walls using different restorative techniques (incremental versus bulk filling) in Class II cavities, and bond strength of silorane- and methacrylate-based restorative systems to dentin after 24 h and 1 year of water storage. The null hypothesis tests were: (1) that there is no difference in polymerization stress between low shrinkage materials and standard composite resins; (2) the type of restorative system and aging time had no influence on internal adaptation; and (3) there is no difference in bond strength of adhesives to dentin when compared to the baseline values and after long-term storage.

2. Material and methods

Four commercial composite resins (shade A2) were evaluated: two low-shrinkage composite resins (Filtek Silorane, 3M ESPE, St. Paul, MN, USA, and Aelite LS, Bisco, Inc., Schaumburg, IL, USA) and two conventional composite resins (Heliomolar and Tetric N-Ceram, Ivoclar Vivadent, Schaan, Liechtenstein) (Table 1). For bond strength test and internal adaptation analysis, three adhesive systems from the same manufacturer at the same composite resins were used: ExciTE (Ivoclar Vivadent, Schaan, Liechtenstein) for Tetric N-Ceram and Heliomolar; One-Step Plus (Bisco, Inc., Schaumburg, IL, USA) for Aelite LS, and Silorane System Adhesive for Filtek Silorane.

2.1. Polymerization stress measurements

Polymerization stress was measured using rods of polymethyl methacrylate (PMMA) as bonding substrate for composite resin, with diameters of 5 mm ($n=10$). Rods were sectioned in 13 and 28 mm segments. For the 13 mm rods, one of the flat surfaces was lapped and polished by hand using #600–1200 sandpaper and felt disks with 1 μm alumina paste (Alumina 3, ATM, Altenkirchen, Germany) to allow for light transmission during photoactivation. One of the flat surfaces of the 28 mm rods was sandblasted with alumina ($250 \,\mu m$) for 10 s at a distance of 1 cm and treated with a methyl methacrylate monomer (JET Acrílico Auto Polimerizante, Artigos Odontológicos

Clássico, São Paulo, SP, Brazil). These surfaces received two thin layers of an unfilled resin (Scotchbond Multipurpose Plus, bottle 3, 3M ESPE, St. Paul, MN, USA), light-activated with 12 J/cm² (600 mW/cm² for 30 s), except for the Filtek Silorane, in which it was applied a thin layer of the Adhesive of Silorane System Adhesive (3M ESPE, St. Paul, MN, LISA).

The rods were attached to the opposite fixtures of universal testing machine (Instron 5565, Canton, MA, USA). On the lower fixture, the 13 mm rod wasfixed to a stainless steel attachment with a slot, allowing positioning of light guide in contact with its polished surface. The 28 mm rod was attached to the upper fixture, which was connected to load cell. The distance between the rods was 1 mm (cavity configuration factor $C=2.5$; volume = 29 mm³). After insertion of composite resin, an extensometer (model 2630-101, Instron, Canton, MA, USA) was attached to rods in order to monitor the distance between them during the test and provide feedback to machine's actuator to re-establish the initial distance. Therefore, the value registered by the load cell corresponded to the force necessary to maintain initial height of specimen in opposition to the force exerted by the polymerization shrinkage of composite. Light-activation was carried out using quartz–tungsten–halogen light-curing unit (VIP Junior, BISCO, Schaumburg, IL, USA). After propagating through the length of the 13 mm rod, the irradiance reaching the composite resin surface was 570 mW/cm². Irradiance was periodically checked with dental radiometer (model 100, Kerr Demetron Corp., Orange, CA, USA). A 20 s exposure for traditional composite resins and 40 s for Filtek Silorane were used, providing radiant exposure of approximately 18 J/cm². The contraction force was monitored for 5 min from the onset of photoactivation and maximum nominal polymerization stress (in MPa) was calculated by dividing the maximum force value by the cross-sectional area of the rod. Statistical analysis was performed with statistical software (MINITAB 15, State College, PA, USA). One-way analysis of variance (ANOVA) for the restorative systems factor was performed, followed by Tukey's post-hoc test $(p < 0.05)$.

2.2. Internal adaptation analysis

Thirty-two freshly extracted caries-free human third molars were selected for this part of the study and stored in a solution of distilled water with thymol 0.2% at $4 \,^{\circ}\text{C}$ for up to 1 month after extractions. Teeth were obtained and used in accordance with protocol approved by the Ethics Committee in Research (90/2009) of School of Dentistry of Piracicaba, State University of Campinas. The cusps were abraded using wet-ground #320-grit silicon carbide paper and then polished using #600-grit. Standardized Class II preparations of the mesial surface of the teeth were made using 3145 diamond burs (KG Sorensen, Cotia, SP, Brazil) with high-speed hand piece (Turbina Extra Torque 605, Kavo do Brasil, Joinville, SC, Brazil) under water irrigation ($n=4$).

Table 1

Materials, manufacturer, composition, and batch number of the composite resins used in this study (information supplied by the MSDS of the manufacturer).

Material (manufacturer)	Composition	Batch number
Heliomolar (Ivoclar Vivadent, Schaan, Liechtenstein)	BisGMA, UDMA, 1,10-decanediol dimethacrylate, camphorquinone, silicon dioxide, ytterbium trifluoride and prepolymerized filler (prepolymers) (46 vol%)	K35053
Tetric N-Ceram (Ivoclar Vivadent, Schaan, Liechtenstein)	Dimethacrylates, additives, catalysts, stabilizers and pigments, barium glass, ytterbium trifluoride, mixed oxide L48183 and prepolymerized filler (prepolymers) (56 vol%)	
Aelite™ LS (Bisco Inc., Schaumburg, IL, USA)	BisGMA, BisEMA, TEGDMA, camphorquinone, glass filler, amorphous sílica (74 vol%)	0900005990
Filtek Silorane (3M ESPE, St. Paul, MN, USA)	Bis-3,4-epoxycyclohexylethyl-phenyl-Methylsilane 3,4 Epoxycyclohexylcyclopolymethylsiloxane, cam- phorquinone, iodonium salt and electrondonor, silanized quartz, yttrium fluoride (55 vol%)	N205711

Abbreviations: bis-phenol A diglycidyl methacrylate (BisGMA), urethane dimethacrylate (UDMA), ethoxylated bisphenol A dimethacrylate (BisEMA) and triethylene glycol dimethacrylate (TEGDMA).

Preparation dimensions were as follows: bucco-lingual width: 4.0 mm; gingivo-occlusal width: 5.0 mm; axial wall: 2.5 mm depth. The occlusal margins were located in enamel and gingival margin was located in dentin. Burs were replaced after three preparations. Cavity preparation was finished with 3145FF (extra-fine) diamond burs (KG Sorensen, Cotia, SP, Brazil) under water refrigeration. The resulting preparations were randomly distributed into 8 groups $(n=4)$ according to the restorative system (Heliomolar/ExciTE, Tetric N-Ceram/ExciTE, Aelite LS/One-Step Plus, Filtek Silorane/Silorane Adhesive System) and the filling technique (incremental oblique and bulk-filling techniques). The adhesive systems were applied following the manufacturer's instructions (Table 2).

To restore the Class II cavity, Omni-Matrix (Ultradent Products Inc., South Jordan, UT, USA) was used. For incremental oblique technique group, composite resins were applied in four increments (\pm 2.0 mm thick each increment) and individually lightactivated (VIP Junior, BISCO, Schaumburg, IL, USA) under irradiance of 600 mW/cm², which was constantly monitored with radiometer, with the distal end of light-curing tip positioned perpendicular to the occlusal surface of the cavity. For bulk-filling group, cavity was filled in a single increment and subjected to light-activated curing for 40 s. Restored teeth were then stored at 37 °C in distilled water for 24 h.

Each restoration was mesio-distally cross-sectioned with diamond blade, obtaining two half parts. Both halves were polished with #600-, #1200-, and #2000-grit SiC papers, followed by diamond pastes (3-, ½-, and ¼-grit) and placed in ultrasonic cleaner (Thornton USC 1400, Unique Group, Indaiatuba, SP, Brazil) for 10 min to remove the polishing debris.

The same prepared restorations were evaluated 24 h and 1 year after polishing. For 1-year analysis, restorations were stored in distilled water at 37 °C in a light-free environment. Impressions of polished surfaces were taken with low-viscosity polyvinyl siloxane material (Express XT, 3M ESPE, St. Paul, MN, USA) and impressions were poured with epoxy resin (Buehler Ltd., Lake Buff, IL, USA). Afterwards, replicas were gold-sputter-coated (Balzers-SCD 050 Sputter Coater, Balzers, Liechtenstein) and observed using SEM (JEOL, JSM-5600LV, Tokyo, Japan) for the evaluation, measurement, and classification of the cavity margins with $200 \times /$ magnification. For each specimen, it was necessary to take approximately 12 images in order to scan the entire perimeter of restoration. For the measurement of marginal gaps, Image J software (National Institute of Health, Bethesda, MD, USA) was calibrated based on the scale bar present in SEM images. This was possible because all photos were taken at the same magnification ($200 \times$). Then the

entire perimeter of the cavity was measured (in mm) to enable the calculation of the percentage of gaps. Gaps were measured and the value converted to a percentage based on the perimeter of each specimen. Statistical analysis was performed with statistical software (MINITAB 15, State College, PA, USA). Three-way analysis of variance (ANOVA) for restorative systems, restorative placement technique, and storage time factors was performed, followed by Tukey's post-hoc test ($p < 0.05$).

2.3. Bond strength test

Thirty-two freshly extracted caries-free human third molars were selected for the study and stored in solution of distilled water and thymol 0.2% at 4 \degree C for up to 1 month after extractions, approved by the Ethics Committee in Research (90/2009) of the Piracicaba Dental School, State University of Campinas. Teeth were then scaled, cleaned, stored in distilled water for 24 h and randomly assigned to four experimental groups according to composite resins $(n=10)$.

Occlusal middle-depth dentin was exposed by sectioning the crowns parallel to occlusal surface with precision low-speed diamond saw (Isomet 1000, Buehler Ltd., Lake Buff, IL, USA) under water-cooling. Dentin standard smear layer was created by polishing the occlusal surface with #600-grit SiC sandpaper for 60 s. Adhesives applied were according to their respective experimental groups (Table 2). Afterwards, clinical crowns were restored with composite resins using three increments of 2.0 mm each. Each increment was light-cured for 20 s (VIP Junior, BISCO, Schaumburg, IL, USA) for Heliomolar, Tetric N-Ceram, and Aelite LS and for 40 s for the Filtek Silorane composite resin, under irradiance of 600 mW/cm², which was constantly monitored with a radiometer. The teeth were stored at 37 °C in distilled water for 24 h.

Restored specimens were then serially sectioned perpendicular to adhesive-tooth interface at 1.0 mm thickness using slow-speed diamond saw. Approximately 4 beams were tested immediately and 4 were stored for 1 year in distilled water, which was changed weekly. Specimens were tested individually by attaching them to a microtensile jig using cyanoacrylate glue (Super Bonder, Henkel/ Loctite, Itapevi, SP, Brazil). Sticks were submitted to a tensile load using universal testing machine (EZ Test, Shimadzu Corp., Kyoto, Japan) at 1.0 mm/min cross-head speed. Digital caliper (Mitutoyo Corp., Kanagawa, Japan) was used to measure the bonding area in square millimeters.

The load in kgf and the bonding surface area of specimens were registered and microtensile bond strengths calculated in MPa.

Table 2

Materials, manufacturer, composition, and batch number of the adhesives used in this study (information supplied by the. MSDS of the manufacturer).

Material (manufacturer)	Composition	Batch number	Directions for uses
One-Step Plus (Bisco, Inc., Schaumburg, IL USA)	Biphenyl dimethacrylate, hydroxyethyl methacrylate, acetone, dental glass	0800005538	Apply phosphoric acid gel 37% to the prepared dentin for 15 s. Remove the gel with a vigorous water spray for 15 s. Remove the excess moisture with foam pellet (wet bonding). Shake bottle once, Apply 2 generous coats to the preparation. Agitate lightly for $10-15$ s. Dry gently for 5 s. Cure for 10 s
Excite (Ivoclar Vivadent, Schaan, Liechtenstein)	Bisphenol A glycol dimethacrylate, ethanol, 2-hydro- xyethyl methacrylate, phosphonic acid acrylate, ure- thane dimethacrylate	L31463	Apply phosphoric acid gel 37% to the prepared dentin for 15 s. Remove the gel with a vigorous water spray for 15 s. Remove the excess moisture with foam pellet (wet bonding). Apply the adhesive on the prepared surfaces for at least 10 s. Dry gently for 5 s. Cure for 10 s
Filtek Silorane System Adhe- sive (3M ESPE, St. Paul, MN, USA)	Self-etching primer: phosphorylated methacrylates, Vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, silane-treated silica filler, initiators, stabilizers Bond: hydrophobic dimethacrylate, phosphorylated methacry- lates, TEGDMA, silane-treated silica filler, initiators, stabilizers	Primer: N209848 Adhesive: N204592	Shake bottle briefly. Apply visibly thick layer. Gentle air dispersion until movement stops. Cure for 10 s Gently dry surface. Apply and leave undisturbed for 10 s. Then, dry for 5 s with maximum air pressure. Cure for 10 s

Statistical analysis was performed with statistical software (MINITAB 15, State College, PA, USA). Two-way analysis of variance (ANOVA) for the restorative systems and storage time factors was performed, followed by Tukey's post-hoc test ($p < 0.05$).

The fractured surfaces of tested specimens were sputter-coated with gold (MED 010, Balzers, Balzer, Liechtenstein) and examined using scanning electron microscope (VP 435, Leo, Cambridge, England). Failure patterns were classified as: Type I – adhesive failure; Type II – mixed failure; Type III – cohesive failure within dentin; and Type IV – cohesive failure within composite resin.

3. Results

3.1. Polymerization stress measurements

Table 3 presents the means of the polymerization stress for the composite resins and the ANOVA-detected statistical difference between them $(p=0.003)$. After five minutes of measuring, the restorative composite resins Tetric N-Ceram and Aelite LS showed lower polymerization stress means than those obtained for the Heliomolar composite resin. Filtek Silorane did not differ among all composite resins.

3.2. Internal adaptation analysis

Table 4 presents the data in percentages of gaps of restoration perimeter. Three-way ANOVA found statistical differences for the following factors: restorative systems ($p < 0.001$), storage time ($p=0.003$), and restorative technique ($p<0.001$). The values of gaps ranged from 4.6% to 66.1% (for composite resins Filtek Silorane and Aelite LS, respectively).

Gap formation was higher for bulk-filling technique than for incremental placement technique, independent of the restorative system tested. The gaps occurred mainly at gingival-axial line angle. The initial percentages of gaps along the perimeter of restorations were lower than those observed after 1 year of storage in distilled water.

The Filtek Silorane composite resin exhibited the lowest percentage of gaps when compared to other restorative systems ([Figs. 1](#page-4-0) and [2\)](#page-4-0), in terms of both evaluation times (24 h and 1 year) and composite resin placement techniques (incremental and bulk) $(p < 0.05)$. The percentage of gap formation for Aelite LS ([Figs. 3](#page-5-0) and [4\)](#page-5-0) composite resin did not differ for Heliomolar ([Figs. 5](#page-6-0) and [6\)](#page-6-0) and Tetric N-Ceram $(p>0.05)$ [\(Figs. 7](#page-7-0) and [8\)](#page-7-0); however, Heliomolar showed higher gap formation than Tetric N-Ceram ($p < 0.05$).

3.3. Bond strength test

[Table 5](#page-8-0) presents the mean bond strengths for the restorative systems after the two storage times. Two-way ANOVA revealed significant differences for the restorative systems factor ($p < 0.001$) and for the storage time factor ($p=0.003$). Initially, Filtek Silorane showed

Table 3 Mean polymerization stresses (standard deviation) of the composite resins used in this study (in MPa).

Means followed by different letters are significantly different.

Table 4

Mean percentages of gaps for restorative systems following the restorative placement techniques and storage times.

Means followed by different letters represent significant differences (3-way ANOVA and Tukey's test, $p < 0.05$). Capital letters compare composite resins within the same technique and time; lower case compare the storage times within the same placement technique and composite resin; symbols (*) represent differences between composite resin placement technique.

the lowest mean bond strength among restorative systems; however, after storage for 1 year, this material showed the highest mean bond strength to dentin. At baseline, Heliomolar/ExciTE, Aelite LS/One-Step Plus, and Tetric N-Ceram/ExciTE restorative systems showed no significant difference in bond strength among them $(p>0.05)$. After storage for 1 year, the Heliomolar/ExciTE restorative system yielded lower bond strength than was observed for Aelite LS/One-Step Plus and Tetric N-Ceram/ExciTE materials ($p < 0.05$). Bond strength of the Filtek Silorane restorative system was unique in that it was not reduced after storage in water for 1 year ($p > 0.05$).

Total-etch adhesives produced a preponderance of mixed failures for both storage times ([Fig. 9](#page-8-0)). At baseline, most of the specimens bonded with silorane self-etching adhesive failed near or at the interface between the adhesive and dentin (Type I). After storage for 1 year, restorative systems presented more cohesive failures in dentin (Type III).

4. Discussion

Regarding polymerization stress, the Filtek Silorane restorative system did not differ from methacrylate-based composite resins (Heliomolar, Tetric N-Ceram, and Aelite LS), thus null hypothesis (1) was accepted. The silorane-based composite resin presented intermediate polymerization stress values, which can be explained by the flexural modulus of the material that is initially high in the pre-gel polymerization $[4,10,13]$. In this phase, the active species presents enough mobility to re-arrange and compensate for the volumetric shrinkage, which would tend to reduce internal and interfacial stresses following the polymerization reaction [\[14,15\]](#page-9-0). Boaro et al. [\[13\]](#page-9-0) also found low volumetric shrinkage (both post-gel and total) for this material, however reported shrinkage stress values compared to methacrylate-based composite resins, as obtained in this study.

For the Aelite LS composite resin, the results suggested that the addition of a high amount of filler is not an efficient approach for reducing the polymerization stress. The high content of filler particles in Aelite LS has somewhat controversial effects on shrinkage patterns. An increase of filler concentration by volume leads to reduction volumetric shrinkage as the resin volume is minimized. However, a high filler volume results in a stiff material with high elastic modulus that cannot efficiently absorb polymerization stresses [\[16\].](#page-9-0)

The results of the present investigation showed that no tested restorative systems (composite resin/adhesive) exhibited gap-free restorations. The continued polymerization shrinkage in association with elastic modulus generates stresses within the material, at the tooth/restoration interface, and within the tooth structure [\[17,18\]](#page-9-0). This stress state is likely to facilitate gap formation, which may reduce the longevity of the restoration $[16]$. Also, the integrity of the bonded interface depends on the interaction between shrinkage, elastic modulus, and adhesion to tooth structure

Fig. 1. Photomicrograph of the internal interface of Class II cavity restored with Tetric N-Ceram using bulk technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

Fig. 2. Photomicrograph of the internal interface of Class II cavity restored with Tetric N-Ceram using oblique incremental technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

[\[19,20\].](#page-9-0) Despite the fact that the manufacturer described the composite resin Aelite LS as being a low-shrinkage composite resin, the high amount of fillers, which did not result in low gap formation when compared to Tetric N-Bond, shows that it is a conventional methacrylate-based composite resin. These results can be explained by their high flexural modulus, which interferes with the polymerization stress [\[13\]](#page-9-0).

Silorane-based restorations showed better internal adaptation than methacrylate-based restorations regardless of the restorative technique used and storage time, and thus null hypothesis (2) was rejected. The decreased gap formation may be associated with the low post-gel shrinkage of these composite resins, which has already been reported $[13,19,21]$ $[13,19,21]$ $[13,19,21]$, and the type of adhesive system used in combination with this composite resin, which contains a self-etch hydrophilic primer and a hydrophobic bonding resin [\[22\].](#page-9-0) Silorane System Adhesive is a two-step adhesive that has been categorized as a mild self-etch adhesive based upon its interaction with dentin up to a depth of a few hundred nanometers [\[22\]](#page-9-0). The primer and adhesive resin adhesive solutions were light-activated separately, which resulted in a typical two-fold bonding layers.

Fig. 3. Photomicrograph of the internal interface of Class II cavity restored with Heliomolar using bulk technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

Fig. 4. Photomicrograph of the internal interface of Class II cavity restored with Heliomolar using oblique incremental technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

Fig. 5. Photomicrograph of the internal interface of Class II cavity restored with Aelite LS using bulk technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

Fig. 6. Photomicrograph of the internal interface of Class II cavity restored with Aelite LS using oblique incremental technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

These layers act as elastic buffer [\[23,24\]](#page-9-0) that tends to compensate the shrinkage stress developed during polymerization reaction of composite resins. The double adhesive layer and the highly hydrophobic nature of Silorane System Adhesive [\[22,25,26\]](#page-9-0) were important not only for obtaining better internal adaptation and dentinal sealing but also to resist hydrolytic degradation during the water-storage for 1 year.

In the present study, filling the cavity using an incremental technique reduced the internal gap formation when compared to a bulkfilling restorative technique. Previous studies using conventional composite resins have already demonstrated that an incremental technique has a beneficial effect on the bond strength [\[7,27,28\]](#page-9-0). This positive effect should be attributed to reduce shrinkage stress by decreasing the C-factor of each layer. The internal adaptation of the rather stiff Silorane composite resin improved with the incremental technique and the curing at the interface level might have been made more efficient by an increase in irradiance power (light is only dimmed by a thin layer of composite resin) and curing time (the total curing time was increased as well). A study by Van Ende et al. [\[29\]](#page-9-0) examined the stress at the adhesive interface with different configuration factors, and their results indicated that cavity configuration affected the microtensile bond strength of the Silorane Adhesive

Fig. 7. Photomicrograph of the internal interface of Class II cavity restored with Filtek P90 using bulk technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

Fig. 8. Photomicrograph of the internal interface of Class II cavity restored with Filtek P90 using oblique incremental technique; (A) image of 24 h of storage and (B) image of 1 year of storage.

System and considered that an incremental layering technique is still required for placement of silorane composite resin restorations.

The high incidence of gaps formation was observed at the gingivalaxial line angles, since there is an accumulation of bonding agents in this line angle, a difficult adaptation of composite resin in this location and tendency of high-stress in these areas [\[30\]](#page-9-0). After 1 year, all restorative systems tested showed significant increases in the gap formation and debonded margins. These gaps may result from waterinduced degradation, which seemed to happen with methacrylatebased composite resin restorations. The composite resin water-induced degradation occurs by water absorption that causes monomer elution [\[31\]](#page-9-0) or by degradation of the silane at the filler-monomeric network interface [\[32\].](#page-9-0) In contrast, silorane-based restorations also had the best marginal stability after 1 year of water storage. The combination of adhesive and composite resin, i.e., the good interaction with dentin and hydrophobicity for silorane, was responsible for such results [\[10](#page-9-0),[11\]](#page-9-0).

Other studies also investigated incidence of gap formation and marginal adaptation and their outcomes [\[33](#page-9-0)–[37\]](#page-9-0) corroborated with this study, which showed good internal and marginal adaptation for silorane restorative system. Mahmoud & Al-Wakeel evaluated the marginal adaptation of ormocer-, silorane-, and methacrylate-based composite restorative systems bonded to dentin cavities immediately after polymerization and after one month and one year of water aging and thermocycling. They found that all composite restorations presented no gap-free margins, but silorane-based system revealed the best marginal adaptation at all aging times [\[33\]](#page-9-0). Another study showed that silorane- and methacrylate-based materials produced restorations with similar immediate interfacial quality, which kept stable after 6 months of water storage, however silorane system showed higher bond strength than the methacrylate restorations as demonstrated in this study [\[36\]](#page-9-0). Ghulman analyzed the effect of cavity configuration on the marginal adaptation of silorane- and methacrylate-based composite resin and concluded that silorane material resulted in better marginal adaptation, but with a tendency to increase the gap formation with C-factor of five [\[34\]](#page-9-0).

Total-etch adhesives exhibited the highest mean bond strength at the baseline, as previously reported [\[38,39\].](#page-9-0) This study found a significant reduction in bond strength for all total-etch adhesives after the aging treatment, evidenced by hydrolytic degradation over time. The weakening of the physical properties of the methacrylate-based resin-dentin-bonded interfaces occurs by chemical reactions of degradation of polymers and exposed collagen fibrils at the base of the hybrid layer [\[40,41\].](#page-9-0)

The dentin bond strength for the Filtek Silorane System Adhesive was the lowest at baseline, but after 1 year of storage in water, it was stable and the highest among the materials. Considering this result, null hypothesis (3) was rejected. The primer solution of Filtek Silorane Adhesive presented a pH 2.7 that

Table 5

Mean bond strengths (standard deviation) after 24 h and 1 year of storage (in MPa).

	Bond strength	
Composite Resin/Adhesive	24 h	1 year
Heliomolar/Excite	51.6 (6.8) Aa	23.1 (4.4) Cb
Aelite LS/One-Step Plus	48.4 (3.9) Aa	27.8 (3.2) Bb
Tetric N-Ceram/Excite	47.0 (2.9) Aa	28.5 (2.7) Bb
Filtek Silorane/Silorane Adhesive	37.3 (4.3) Ba	36.1 (2.1) Aa

Means followed by different letters (uppercase-column; lower case-row) are significantly different.

provided a mild superficial demineralization of the tooth structure and the functional monomer seem to present chemical bonding to the hydroxyapatite crystals [\[22\]](#page-9-0), The hydrophobicity of the adhesive system may endow bonding layer hydrolytic-resistant characteristics with low water sorption [\[22\]](#page-9-0), leading to a mechanical stability of the adhesive interface [\[41,42\]](#page-9-0).

The failure patterns related for each group depended on the adhesive system used or their interaction with the composite resin (Fig. 9). Initially, the experimental groups that used total-etch adhesives showed mixed failures. For all materials, after 1 year of water storage, a slightly higher percentage of cohesive failure in the dentin and composite resin (Types III and IV) was reported, suggesting the degradation of the collagen matrix of dentin or the monomeric components of the composite resins that occurred in large numbers in the group using Heliomolar composite resin. Most Filtek Silorane specimens failed near the bonded interface between the adhesive layer and composite resin, suggesting that this interface is the weakest link. This finding is a reminder of the difference in nature between the silorane composite resin and the adhesive that connects it with the tooth structure.

`Further in vitro studies that simulate clinical conditions and clinical trial investigations are required to ensure the adequate clinical performance of silorane-based restorative materials, which seem to be a very promising material in terms of its mechanical/ chemical properties, however the viscosity and handling of this material is still a clinical challenge and also a challenge for the manufacturers.

5. Conclusion

Within the limitations of this study, the following conclusions were drawn:

- 1. The shrinkage stress findings describe that silorane-based composite resin can be used for the same type of restorations that the methacrylate ones have been used.
- 2. The best adaptation to the cavity walls promoted by silorane restorative system is important for the internal and marginal sealing, which is related to the longevity of the restorations.
- 3. The mean bond strength for the silorane-based composite resin remained stable after one year of storage in distilled water, which suggest that longevity of silorane restorations is very promising.

Fig. 9. Failure modes of experimental groups.

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