Reparability of Aged Silorane With Methacrylate-Based Resin Composite: Micro-Shear Bond Strength and Scanning Electron Microscopy Evaluation

L Giachetti • D Scaminaci Russo • M Baldini
C Goracci • M Ferrari

Clinical Relevance

Aged silorane composite restorations can be repaired with a methacrylate-based resin composite by using a phosphate-methacrylate–based adhesive as the intermediate layer.

*Luca Giachetti, MD, DMD, MSc, Tuscany School of Dental Medicine, University of Florence and Siena, Italy, Department of Public Health, Dentistry, University of Florence

Daniele Scaminaci Russo, DDS, Tuscany School of Dental Medicine, University of Florence and Siena, Italy, Department of Public Health, Dentistry, University of Florence

Matteo Baldini, DDS, Tuscany School of Dental Medicine, University of Florence and Siena, Italy, Department of Public Health, Dentistry, University of Florence

Cecilia Goracci, DDS, MSc, PhD, Tuscany School of Dental Medicine, University of Florence and Siena, Italy, Department of Dental Materials and Fixed Prosthodontics, University of Siena

Marco Ferrari, MD, DMD, PhD, Tuscany School of Dental Medicine, University of Florence and Siena, Italy, Department of Dental Materials and Fixed Prosthodontics, University of Siena

* Corresponding author: Viale Morgagni 85, Firenze, 50134 Italy; e-mail: luca.giachetti@unifi.it

SUMMARY

Objectives: To evaluate the compatibility between aged siloranes and methacrylate-based composites by simulating a common repair-technique.

Methods: Twenty substrates were constructed using silorane (Filtek Silorane, 3M ESPE) and methacrylate composites (Filtek Supreme XT, 3M ESPE). Substrates were aged in 0.9% NaCl solution at 37°C for 72 hours. Silorane build-ups were constructed on silorane substrates without any intermediate layer (IL). Methacrylate build-ups were constructed on silorane substrates without any IL, with a methacrylate IL (Heliobond, Ivoclar Vivadent), or with a phosphate-methacrylate IL (Silorane System Adhesive Bond, 3M ESPE). Methacrylate build-ups were also constructed on methacrylate

substrates without any IL. The micro-shear bond strength test was carried out after thermocycling. Bond strength data were statistically analyzed using analysis of variance and Tukey post hoc tests. Failure modes were assessed by means of scanning electron microscopy observations.

Results: The silorane-methacrylate group without any IL showed the lowest bond strength values (0.4 ± 0.1 MPa). The use of a methacrylate-based IL (1.6 ± 1.7 MPa) led to a slight increase in bond strength, whereas the use of phosphate-methacrylate IL (9.1 ± 5.4 MPa) significantly increased bond strength. There was no statistically significant difference in bond strength between silorane-silorane (7.9 ± 3.6 MPa) and methacrylate-methacrylate (9.5 ± 4.1 MPa) groups without any IL.

INTRODUCTION

The use of composite resin restorative materials has been widely accepted in dental practice.[1] However, although methacrylate-based composites exhibit acceptable clinical performance, polymerization shrinkage is still a drawback.[2] Polymerization shrinkage results in volumetric contraction, causing stress in bonded restorations that can lead to clinical failure.[3-7] Recently, a new category of polymers for dental-restorative use was introduced: silorane-based composites. Polymerization of silorane-based composites occurs through a photocationic ring-opening reaction, which results in a lower polymerization contraction compared with the free radical polymerization of dimethacrylate monomers.[8,9] The volumetric shrinkage of the silorane composite was determined to be 0.9%, which is clearly the lowest value observed for the investigated materials.[9] This is in good agreement with stress measurements by Ernst and others,[10] showing the lowest stress development for siloranes among all tested composite materials.

According to the manufacturer of Filtek Silorane (3M ESPE, St Paul, MN, USA), which is the only silorane-based marketed composite, silorane composite resin can be used for direct class I and class II restorations and as a base under a methacrylate-based composite. In fact, siloranes can have an important role as a base under methacrylate composite resin in what is commonly referred to as a sandwich restoration. By replacing part of the methacrylate composite with silorane composite, it is possible to obtain lower shrinkage and consequent-ly lower polymerization stress. In addition, more esthetically satisfactory results could be achieved by using methacrylate composite resin as an enamel restorative. According to the literature,[11,12] in order to stratify a methacrylate composite on a silorane composite, the use of a phosphate-methacrylate–based intermediate resin such as the second component (Bond) of the Silorane Adhesive System (3M ESPE) is required. The application of this hydrophobic resin coating promotes bonding not only to silorane-based composites but also to methacrylate-based materials.[13] The phosphate group reacts with oxirane, whereas the acrylate group reacts with dimethacrylate, thus resulting in the adhesion between the two composites.[11] Tezvergil-Mutluay[11] also demonstrated that the bond strength between consecutive layers of silorane composite decreased when the time of placement between consecutive layers increased. This suggests that as the chemical reactivity decays over time,[14] the bond strength could be affected. Consequently, another clinically interesting point to consider is the possibility of repairing an aged silorane restoration with a conventional methacrylate composite system. According to the manufacturer, silorane restorations can be repaired with a conventional methacrylate composite system using a dimethacrylate-based intermediate layer. On the other hand, according to the literature,[11,12] the use of a phosphate-methacrylate–based adhesive as an intermediate layer could be more appropriate. However, the studies by Tezvergil-Mutluay and others[11] and Lührs and others[12] were conducted on fresh substrates, while it has not yet been determined whether a phosphate-methacrylate–based adhesive should also be applied as an intermediate layer on aged silorane composite restorations.

Using the micro-shear bond strength test (μSBS) and scanning electron microscopy (SEM), the aim of this study was to evaluate the ability of silorane- and methacrylate-based aged composites to be repaired and to examine the compatibility between siloranes and methacrylate-based composites by simulating a common repair technique. The tested null hypotheses were 1) there is no difference in bond strength between silorane-silorane, methacrylate-methacrylate, and silorane-methacrylate combinations, and 2) similar bond strength develops between silorane-based and methacrylate-based composites regardless of the application of an intermediate bonding layer.

MATERIALS AND METHODS

The materials used in this study are listed in Table 1. A silorane composite (Filtek Silorane, A3 shade,

Table 1: *Chemical Composition and Batch Numbers of the Tested Materials*

Materials	Manufacturer	Batch No.	Material Composition
Filtek Silorane	3M, ESPE	7AU	1,3,5,7-Tetrakis (ethyl cyclohexane epoxy)
			1,3,5,7-tetramethyl cyclotetrasiloxanemethyl-bis[2-(7-oxabicyclo[4.1.0]hept-3-yl)ethyl]phenyl
Filtek Supreme	3M, ESPE	6FK	bis-GMA, Bis-EMA, TEGDMA, UDMA
Silorane System Adhesive Bond	3M, ESPE	7AJ	TEGDMA, Phosphoric acid methacryloxyhexylesters, 1,6-hexanediol dimethacrylate
Heliobond	Vivadent-Ivoclar	405316	bis-GMA, TEGDMA

Abbreviations: bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate; bisGMA, bisphenol A-glycidyl dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.

3M ESPE, St Paul, MN, USA) and a methacrylate composite (Filtek Supreme XT, A3B shade, 3M ESPE) were used as substrate and adherent materials. As a substrate material, silorane was used in groups 1 to 4, whereas a methacrylate-based composite was used as a substrate in group 5 (Table 2). The substrates were fabricated by placing unpolymerized composite between two glass microscope slides. The material was then light polymerized for 30 seconds with a light-curing device (Astralis 10, High Power Program 1200 mW/cm^2, Vivadent-Ivoclar, Schaan, Liechtenstein). Thus, disks of about 15 mm in diameter and 1 mm in thickness were obtained. A 2-mm-diameter hole was produced near the margin of the disk using a hand piece and a parallelometer (CL-MF2002S, Heraeus-Kulzer Inc,

Hanau, Germany) in order to allow for repeatable placement of the disk during the mechanical test. Substrates were aged in 0.9% NaCl solution in a light-proof container at 37°C for 72 hours and then randomly divided into five groups (n=4) according to the used method of substrate preparation (Table 2). One of the two surfaces of each disk was roughened for five seconds with P600-grit abrasive paper (WS Flex 18 C, Hermes Abrasives Ltd, Virginia Beach, VA, USA) under running water using a lapping machine (LS2, Remet, Bologna, Italy). Silorane build-ups were constructed on silorane substrates without any intermediate layer (IL; group 1). Methacrylate build-ups were constructed on silorane substrates without any IL (group 2), with a methacrylate IL (Heliobond, Ivoclar Vivadent; group 3) or

Table 2: *Descriptive Statistics of Micro-Shear Bond Strength and Distribution of Failure Modes[a]*

Group	Substrate	Intermediate Layer	Build-up	Number of Tested Specimens	Bond Strength (MPa) Mean ± SD	Fracture Mode		
						Adhesive	Cohesive	Mixed
1	F.Silorane	/	F.Silorane	27	7.9 ± 3.6[a]	12 (44%)	15 (56%)	—
2	F.Silorane	/	F.Supreme	4	0.4 ± 0.1[b]	4 (100%)	—	—
3	F.Silorane	Heliobond	F.Supreme	25	1.6 ± 1.7[b]	25 (100%)	—	—
4	F.Silorane	Silorane System Adhesive Bond	F.Supreme	30	9.1 ± 5.4[a]	19 (63%)	6 (20%)	5 (17%)
5	F.Supreme	/	F.Supreme	27	9.5 ± 4.1[a]	5 (18%)	22 (82%)	—

[a] In the Bond Strength column, different superscript letters label statistically significant between-group differences (p<0.05).
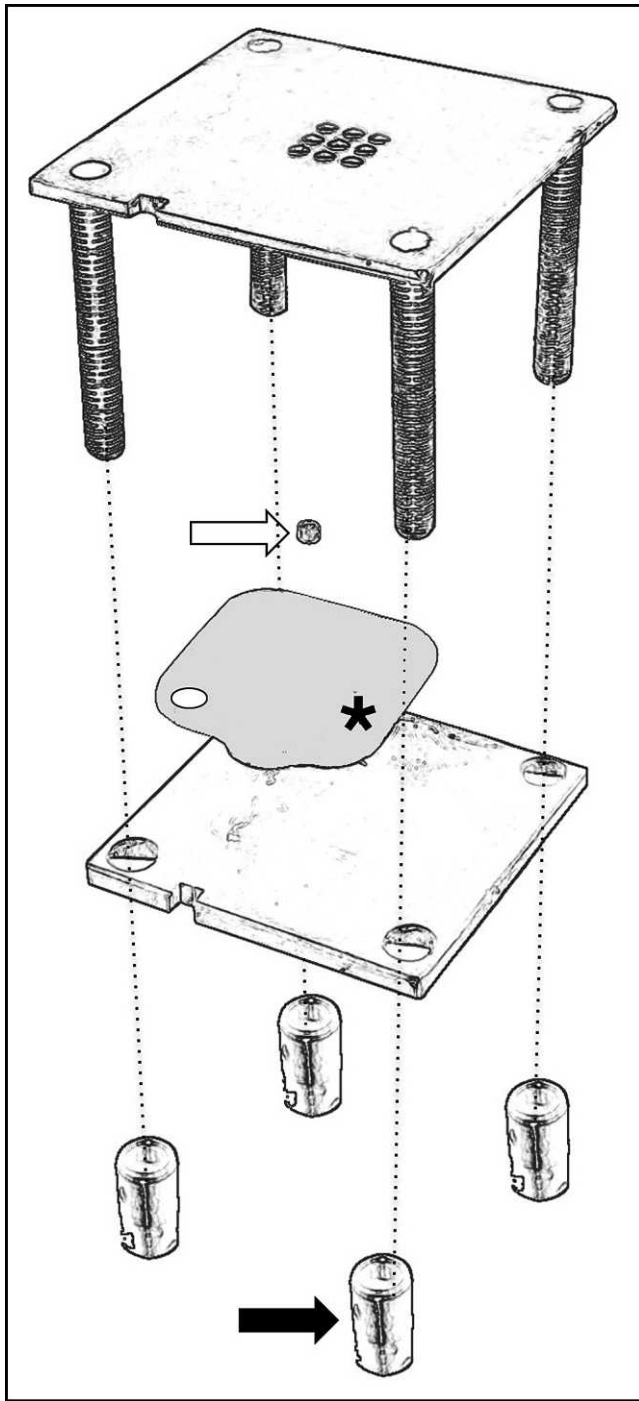
Figure 1. *Metal device for specimen construction. Silicone tubules (blank arrow) were embedded into the lower sections of the holes. A resin composite disc (asterisk) was placed between the two plates, which were then joined together with four screws (black arrow).*

with a phosphate-methacrylate IL (Silorane System Adhesive Bond; group 4). Methacrylate build-ups were also constructed on methacrylate substrates without any IL (group 5). The latter group was tested as a control.

The methacrylate IL and the silorane IL were applied to one surface of each disk by means of a micro brush, thinned with a light air stream, and light polymerized for 20 seconds.

The build-ups were constructed using a custom-made device (Figure 1), consisting of two parallel metal plates that could be joined by means of four screws. The upper plate had nine holes. Each hole was made of two sections: a lower section (1.7 mm in diameter) and an upper section (1.45 mm in diameter). Nine silicone tubules having an internal diameter of 0.7 mm, an external diameter of 1.7 mm, and a height of 0.5 mm were custom made from a silicone tube (art.30/07; Stonfo, Florence, Italy). The tubules were embedded into the holes of the lower section. Each resin substrate was placed between the two plates, which were then joined together with the four screws. The silicone tubules were then filled with the resin composite, which was subsequently light cured for 20 seconds. After opening the device, the silicone tubules were easily removed from the build-up (Figure 2). The built-up specimens were stored in 0.9% NaCl solution in a light-proof container at 37°C for 24 hours and then thermo-cycled (1500 cycles between 5°C and 55°C; 10-second dwell time in each 0.9% NaCl bath; LTC, LAM Technologies, Firenze, Italy). The specimens were observed under a stereomicroscope (40× magnification; SMZ-10, Nikon Corporation) to verify integrity at the build-up/substrate interface. Build-ups that showed apparent interfacial gap formation, bubble inclusion, or any other relevant defect were excluded from the study.

Specimens were subjected to a µSBS test using a universal testing machine (LMT-100, LAM Technologies). The specimen was placed on the testing machine by positioning the hole in the specimen around a pin on the moving part of the machine. The hole could freely rotate around the pin. A thin stainless-steel wire (diameter = 0.20 mm) was looped around a pin on the stationary part of the machine as well as around the resin build-up. The wire contacted half the circumference of the build-up and was gently held flush against the disk at the build-up–substrate interface. Thus, the wire loop exerted shear forces parallel to the bonded interface, at a crosshead speed of 1 mm/min until failure occurred. The µSBS was expressed in MPa by dividing the load at failure (N) by the surface area (mm^2).

Statistical Analysis of µSBS Data

As the data distribution was normal in each group (Kolmogorov–Smirnov test) and group variances
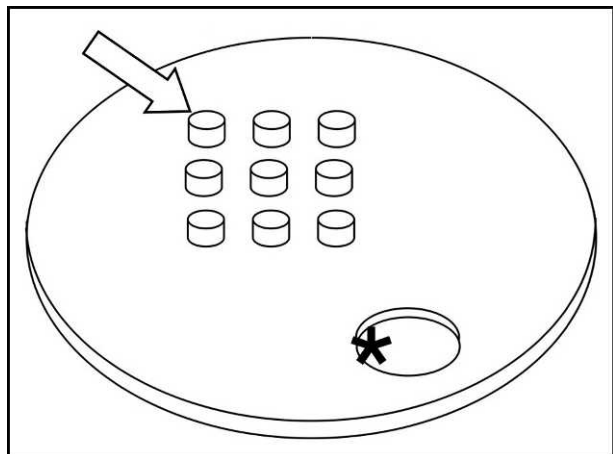
Figure 2. *A schematic representation of a specimen showing the hole near the margin of the resin composite disc (asterisk) and resin composite build-ups (blank arrow).*

Figure 3. *The means and standard deviations of micro-shear bond strengths in different groups. Group 1: Silorane + Silorane. Group 2: Silorane + Supreme. Group 3: Silorane + Heliobond + Supreme. Group 4: Silorane + Silorane System Adhesive Bond + Supreme. Group 5: Supreme + Supreme.*

were homogeneous (Levene test), the one-way analysis of variance (ANOVA) was applied, followed by the Tukey test for post hoc comparison. Between-group differences in the distribution of failure modes were statistically assessed using chi-square tests. In all the analyses, the level of significance was set at $p < 0.05$ (SigmaStat 3.5 Statistical Software package, Systat Software Inc, San Jose, CA, USA).

Specimen Preparation for SEM Observation

Following the µSBS test, the substrates and the debonded build-ups were prepared for SEM analysis. The specimens were mounted on aluminum stubs with colloidal silver paint and sputter coated (SCD 005, BAL-TEC AG, Balzers, Liechtenstein) with 200-Å gold-palladium alloy (Foil Target AU, BAL-TEC AG). To determine the mode of failure, each specimen was observed under the SEM (Philips 515, Philips Co, Amsterdam, The Netherlands; 15-KV accelerating voltage) at 100× magnification. Images were acquired by means of a computerized program (Analysis 2.1, Soft Imaging System GmbH, Munster, Germany).

RESULTS

This study involved the preparation of nine build-ups on each substrate. Of 180 build-ups, 4 from group 1, seven from group 2, three from group 4, and three from group 5 either prematurely failed or accidentally detached from their base after opening the device. Under stereomicroscope observation, five build-ups from group 1, six build-ups from group 2, three build-ups from group 3, three build-ups from group 4, and six build-ups from group 5 showed
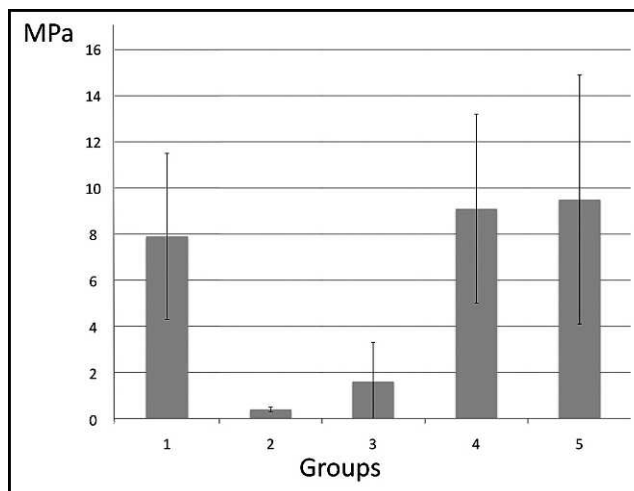
defects at the adhesive interface and were discarded. Nineteen build-ups from group 2 and eight build-ups from group 3 failed during thermocycling. Consequently, 127 build-ups (27 build-ups from group 1, four build-ups from group 2, 25 build-ups from group 3, 30 build-ups from group 4, and 27 build-ups from group 5) were subjected to µSBS testing.

µSBS Test

Descriptive statistics of µSBS are reported in Table 2 and Figure 3 along with statistically significant between-group differences. Measured bond strengths were (mean ± standard deviation) 7.9 ± 3.6 MPa for group 1, 0.4 ± 0.1 MPa for group 2, 1.6 ± 1.7 MPa for group 3, 9.1 ± 5.4 MPa for group 4, and 9.5 ± 4.1 MPa for group 5. The one-way ANOVA showed that groups differed significantly ($p < 0.001$). In particular, the post hoc test revealed that the bond strengths measured in groups 1, 4, and 5 were significantly higher than those recorded in groups 2 and 3.

SEM Observation

SEM observation showed different fracture patterns among the groups (Figure 4).

All specimens of groups 2 and 3 failed adhesively. Both adhesive (44%) and cohesive (56%) failures were noticed in group 1, while failures were mostly adhesive (63%) in group 4 and mainly cohesive in group 5 (82%).
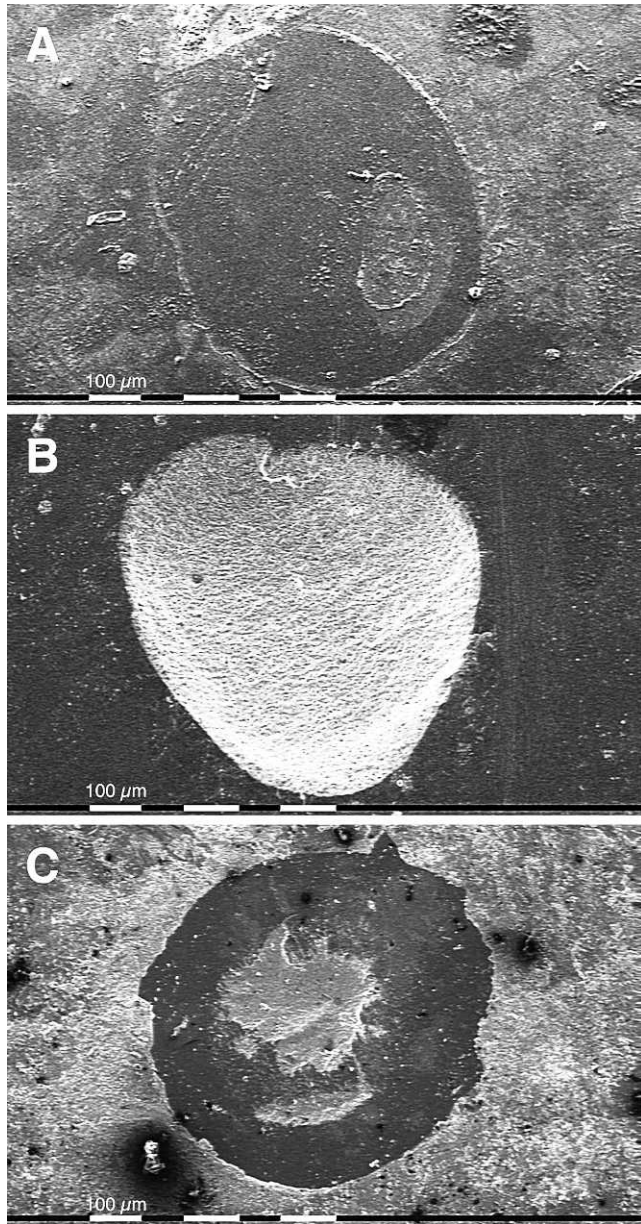
Figure 4. *Scanning electron microscopy microphotographs of the debonded surface of tested substrates (100×). (A): Adhesive failure in a silorane-silorane specimen (group 1). (B): Cohesive fracture of a methacrylate build–up, which was constructed on a methacrylate substrate (group 5). (C): Mixed fracture of a silorane build-up created on a methacrylate substrate with the interposition of a phosphate-methacrylate intermediate layer (group 4).*

The differences in failure mode distribution were statistically significant, with the exception of the comparison between groups 2 and 3, both of which exhibited exclusively adhesive failures.

DISCUSSION

This study was designed to evaluate the ability of an aged silorane-based composite to be repaired with methacrylate-based composite by simulating a common repair technique. Based on the study's findings, the first null hypothesis has to be rejected. As a matter of fact, the silorane-methacrylate group without any intermediate layer (group 2) exhibited significantly lower bond strengths than those recorded by the silorane-silorane and the methacrylate-methacrylate groups without adhesive layer (groups 1 and 5, respectively). Also, in group 2, the highest number of failures during thermocycling was recorded, and all the tested specimens failed adhesively at the silorane-methacrylate interface. No statistically significant difference in bond strength emerged between silorane-silorane (group 1) and methacrylate-methacrylate (group 5) groups. However, the two groups exhibited significantly different failure patterns. Specifically, group 5 had a predominance of cohesive failures. Also, the second formulated null hypothesis has to be rejected because the use of the phosphate-methacrylate–based adhesive as IL (group 4) significantly increased the bond strength. Statistically similar adhesion levels were instead obtained either with or without the application of a methacrylate-based IL (groups 3 and 2, respectively). The application of the phosphate-methacrylate–based IL also had an influence on specimen failure mode. It was indeed only with the interposition of this bonding material that mixed and cohesive failures occurred in silorane-methacrylate combinations. Therefore, according to the results of this study, the application of the phosphate-methacrylate–based adhesive as IL is beneficial when repairing an aged silorane-based composite with a conventional methacrylate-based composite.

The ring-opening reaction of the silorane is a cationic polymerization reaction, and no oxygen inhibition occurs on the polymerized surface.[11] Therefore, the bond between consecutive layers depends on the reactivity of the material.[11] Chemical reactivity is known to decrease over time.[14] As a result, the time interval between placement of consecutive silorane layers must not be overly long. A five-minute delay between layers results in significantly decreased bond strengths and increased percentage of adhesive failures.[11] However, the results of this study showed that the bond strength of an additional layer of silorane composite added to an aged silorane substrate is comparable to the bond strength of a layer of methacrylate composite added to an aged methacrylate resin substrate. On the other hand, the fact that the specimens in group 2 showed the lowest bond strength and the highest number of premature failures, when compared with

the other groups, suggests that there is no chemical compatibility between the aged silorane substrate and the added methacrylate resin composite. However, the use of Silorane System Adhesive Bond as an intermediate layer between the aged silorane composite and a methacrylate composite led to a significant increase in bond strength. Silorane IL is based on methacrylate chemistry with the addition of phosphate groups. The reaction of the phosphate group with oxirane and of the acrylate group with dimethacrylate might account for the recorded increase in bond strength.[11]

Numerous repair modalities have been evaluated *in vitro* for conventional methacrylate-based composites.[15-19] The treatment of methacrylate composite surfaces with a methacrylate bonding agent can be regarded as a standard procedure in today's dentistry. On the other hand, this study demonstrated that the application of a methacrylate-based IL is not beneficial when repairing an aged silorane-based composite with a conventional methacrylate-based composite. Therefore, it can be hypothesized that the repair modality for a silorane-based composite is different from that of a methacrylate-based resin composite. However, in clinical practice, the operator is blind to the type of composite resin that was originally used to restore the tooth. According to the literature,[20] Silorane System Adhesive can also be used to bond conventional methacrylate-based composites to dentin. However, the application of the Silorane System Adhesive Bond as an intermediate layer when repairing an aged methacrylate-based composite with a fresh methacrylate-based composite has not yet been tested. Further research should be carried out to allow clinicians to use Silorane System Adhesive Bond as an IL in reparations regardless of the type of composite used in the original restoration.

A μSBS test was carried out to measure the bond strength between silorane-based and methacrylate-based resin composites. This test represents a viable screening mechanism for predicting clinical performances and allows easier sample preparation as compared with other bond strength evaluation methods.[21] However, the bond strengths measured in this study were significantly lower when compared with bond strengths resulting from previous micro-shear bond test studies.[22-26] The sample preparation required for the μSBS test is not clearly and extensively described in literature.[23-34] In particular, it is not clear how the silicon tubules were held firmly on the dentin surface to prevent the resin from seeping away from the defined area at the base of the cylinder. Consequently, to avoid such inconvenience, samples were prepared by means of an especially designed, custom-made device. The lower bond strength measured in this study can be attributed to this newly devised method of sample preparation. In this study, the μSBS test was preferred to the microtensile test as it allowed easier quantification of the number of specimens that prematurely failed or accidentally detached during preparation. The number of prematurely failed, discarded specimens in each test is probably related to the aggressiveness of the preparation procedure.[35] The cutting procedure that is carried out during the microtensile test transmits vibrations to the specimens. Consequently, a common occurrence, especially if the bond strengths are relatively low (5–7 MPa),[36] is a premature failure of the specimen, which makes microtensile useless.[37-39] In this regard, because of the weak bond strength between silorane-based and methacrylate-based resin composites, the micro-shear test was preferred to microtensile as it did not require cutting after bonding, which avoided any additional stress on specimens.

CONCLUSION

A reliable bond between aged silorane composite and methacrylate composite was obtained by using a phosphate-methacrylate–based adhesive as an intermediate layer. The interfacial bond strength achieved when repairing aged silorane with silorane was similar to that obtained by repairing aged methacrylate-based composite with methacrylate-based composite.

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