Effect of Different Surface Treatments and Adhesives on Repair Bond Strength of Resin Composites After One and 12 Months of Storage Using an Improved Microtensile Test Method

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Clinical Relevance

Repairing and extending composite restorations is enhanced by mechanical roughening and applying freshly made silane on the old composite filling and the use of an adhesive rendering a thin bonding layer.

SUMMARY

Objectives: To evaluate the effect of surface treatments and bonding systems on the repair bond strength between composite materials after one and 12 months of storage, using an improved microtensile test method.

Methods: A total of 72 composite cylinders (Tetric Evo Ceram, Ivoclar) were fabricated,

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stored in distilled water for two weeks followed by thermal cycling (5000 times between 5°C and 55°C), and served as substrate. The cylinders were mechanically roughened using 320-grit silicon carbide sandpaper, etched with 37% phosphoric acid gel, rinsed with water, and divided equally into three experimental groups: group 1, unchanged surface; group 2, sandblasting of the surface (CoJet tribochemical silica sand, 3M ESPE; Microetcher II, Danville Engineering Inc); and group 3, surface silane coating (Bis-Silane, BISCO Inc). Eight control cylinders were prepared and underwent similar aging as the substrate. Each experimental group was divided into subgroups that received the following bonding systems: one-step self-etching adhesive (AdheSE One, Ivoclar Vivadent), two-step self-etching adhesive (Clearfil SE, Kuraray America),

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and three-step etch-and-rinse adhesive (Adper Scotchbond Multi-Purpose, 3M ESPE). Fresh composite (Tetric Evo Ceram, Ivoclar) was placed and cured on top of the prepared substrate cylinders. The specimens were placed in distilled water for a week and thermocycled the same way as before. Eight composite control cylinders were also stored and thermocycled for the same period of time. Half of the cylinders in each test group were tested at one month and the second half at 12 months. The cylinders were serially sectioned in an automatic cutting machine, producing 10 to 20 1.1×1.1 -mm test specimen beam from each cylinder. Specimens were prepared for microtensile testing and the tensile strength calculated based on the force at fracture and specimen dimension. The fracture surfaces were examined under a stereomicroscope and the type of fracture noted.

Results: The mean tensile strength of composite control was 54.5 \pm 6.0 MPa at one month and 49.6 \pm 5.1 MPa at 12 months. The mean tensile strength for the repaired groups ranged from 26.4 \pm 6.8 MPa to 49.9 \pm 10.4 MPa at one month and 21.2 \pm 9.9 to 41.3 \pm 7.5 at 12 months. There was a statistical difference between all groups (p < 0.05) at one month. This difference was less pronounced at 12 months. The highest repair strength was obtained in the group having a silane-coated surface and Clearfil, the two-step self-etching adhesive. Clearfil also had the highest repair strength within each surface treatment group. There was a tendency for lower tensile strength at 12 months compared with one month. Most fractures were of the adhesive type; the highest number of cohesive fractures, 16% at one month and 12% at 12 months, were in groups with the highest tensile strength.

Conclusion: The best repair bond strength was achieved by using freshly mixed silane solution on the substrate in addition to an adhesive, rendering a thin bonding layer.

INTRODUCTION

The replacement of failed restorations is a major dental health care expense and accounts for roughly half of restorative dental work.¹⁻⁴ Removing faulty bonded-composite restorations is a demanding and time-consuming task. It has been demonstrated in a clinically simulated study that more than twice as much tooth structure was lost when removing

composite restorations than comparable amalgam restorations.⁵ As a consequence, a more conservative and minimally invasive approach—repair rather than replacement of the whole restoration—has been suggested when possible.^{2,6,7} This approach and philosophy has gradually been adopted by most Western dental schools.⁸⁻¹² Furthermore, some clinical evidence has been presented that repairing composites increases the longevity of the restorations.¹³

Since the introduction of resin composite materials, researchers have explored methods to repair composite restorations by adding new composite to the old. ¹⁴ New composite can possibly be retained to old composite either through chemical bonding to the filler particles and the organic matrix or through micromechanical bonding to irregularities in the prepared surface. 15 It is, however, generally accepted that as composite ages and water uptake occurs, there is a significant reduction in available carboxyl double bonds for chemical polymerization to new composite. 16-18 In the last two decades various reports have been published on the repair strength of resin composites. The vast majority of these investigations deal with different surface treatments of the original substrate to be repaired. This includes mechanical roughening with various grits of diamond burs and sandpaper, 19-31 abrasion with pumice,³² sandblasting with aluminum oxide or silicatized sand,^{22,24-42} etching with various concentrations of hydrofluoric acid,^{22,25-27,31,41,43-45} application of 38% hydrogen peroxide,³⁵ and silane application. ^{19,26,28,30,35,43,46,47} In all these investigations some adhesive was used as a wetting agent in the repair process, in addition to using only flowable composite as an adhesive³⁶ and preheating the repairing composite.³⁴ In the majority of these studies, except in some using shear bond testing, the adhesive testing is performed shortly after the repair process without aging the repaired specimens. From none of these studies has a preferred method emerged to repair bisphenol A glycidyl methacrylate (Bis-GMA)-based composite resin restorations.

Storage in water for different periods is the most common procedure to age the composite to be repaired, and in a few studies thermal cycling, using extreme mouth temperatures, has been applied. ^{23,48} Some less clinically oriented aging methods have also been used, like boiling the specimens for several hours or immersing them in citric acid for a week. ⁴⁸

Most of the recent studies on repair of composites have examined the repairability of silorane-based restoratives. 49-52 In one recent study, however,

Product	Manufacturer	Lot No.	Expiration Date	
Tetric Evo Ceram caps, shade B2	Ivoclar Vivadent AG, 9494 Schaan, Liechtenstein	N70113	2014-01	
Tetric Evo Ceram syringe, shade A2	Ivoclar Vivadent AG, 9494 Schaan, Liechtenstein	P02083 and P11483	2014-12	
AdheSE One F	Ivoclar Vivadent AG, 9494 Schaan, Liechtenstein	N58194	2012-09	
Clearfil SE Bond	Kuraray America Inc New York, NY 10038, USA	Primer: 01043A	2013-04	
		Bond:01557A	2013-04	
Adper Scotchbond Multi-Purpose	3M ESPE Dental Products, St Paul, MN	Etch: N231977	2014-01	
	55144-1000, USA	Primer: N236935	2014-01	
	_	Adhesive: N229564	2013-11	
Bis-Silane	BISCO Inc, Schaumburg, IL 60193, USA	Part A: 1000008430	2012-07	
		Part B: 1000008431	2012-07	
CoJet system	3M ESPE Dental Products, St Paul, MN 55144-1000, USA	CoJet sand: 355331	2012-04	

repairability of many different composites was investigated.²⁵ It was concluded that none of the surface treatments tested could be recommended as a universally applicable repair technique. The effect of adhesive layer thickness on bond strength between composite and dentin has been investigated,⁵³⁻⁵⁶ but no work on the effect of the thickness of the adhesive layer when repairing composite could be located.

The main objective of this *in vitro* investigation is to evaluate the repair bond strength between composite materials using microtensile testing of specimens united by different bonding systems and surface treatments. In addition to three different bonding systems representing self-etch and three-step etch-and-rinse systems, three different surface pretreatments¹⁰⁻¹² were used. The tested null hypotheses were as follows: 1) the repair bond strength is independent of the type of the bonding system; 2) the repair bond strength is independent of surface pretreatment; 3) the durability of the repair bond strength decreases over the course of time.

METHODS AND MATERIALS

All the restorative materials used in this study are listed in Table 1. The procedure and preparation of the composite cylinders is summarized in Table 2. A total of 72 Tetric Evo Ceram (Ivoclar Vivadent, Schaan, Liechtenstein) composite cylinders, 10 mm in diameter and 6 mm in height, were fabricated in Teflon molds. The composite cylinders were incrementally built in three layers and each layer cured for 40 seconds with a Demetron A2 corded LED curing light (Kerr Corp, Orange, CA, USA). The light output was measured at 1100 mW/cm² (Norwegian Radiation Protection Authorities, Österaas, Nor-

way). A Mylar strip and glass slide was used at both ends of the Teflon mold to achieve flat-ended specimen blocks. In addition, as a control group, eight composite cylinders of the same diameter and 12 mm in height were incrementally fabricated.

After polymerization, the cylinders were immediately stored in distilled water for a total of two weeks. ⁵⁷ The cylinders were further aged by thermal cycling 5000 times between 5°C and 55°C, with a dwell time of 20 seconds and transfer time of three seconds. The 72 cylinders were all surfaced flat with a 320-grit silicon carbide sandpaper disc (Struers, Copenhagen, Denmark) under running water for 5 seconds to obtain a flat surface with standardized roughness.

For cleaning purposes, all the experimental composite cylinders were acid etched with 37% phosphoric acid gel for 15 seconds and rinsed with water for another 15 seconds. The aged cylinders were randomly divided into three experimental groups to have the following surface treatments: Group 1— The 320-grit sandpaper finish unchanged. Group 2— The cylinders were coated with CoJet tribochemical silica sand (3M ESPE, St Paul, MN, USA) using an intraoral sandblaster (Microetcher II, Danville Engineering Inc, San Ramon, CA, USA) for 20 seconds at a distance of about 5 mm. Residual sand was removed by a stream of air for five to 10 seconds. Group 3—The cylinders were coated with Bis-Silane (BISCO Inc, Schaumburg, IL, USA) two-part silane porcelain primer. The two parts were mixed and applied to the test surfaces with a small brush for 30 seconds and gently dried with air for five to 10 seconds to evaporate the solvent. A fourth group contained the eight control cylinders.

Base specimens	Tetric Evo Ceram shade B2 cylinders (Diam: 10 mm, height 12 mm)	Tetric Evo Ceram shade B2 cylinders (Diam:10 mm, height 6 mm)									
Aging	Wa	ater storage a	er storage and thermo cycling [5000X (5 and 55 °C, dwell time: 20 sec, transfer time 3 sec)] total 14 days								
Surface treatment 1			Mechanical roughening with sandpaper, 320 grid								
Rinsing			Acid etch (37% phosphoric gel for 15 sec) + water rinse (15 sec)								
Surface treatment 2			Non			CoJet			Bis-Silane		
Bonding procedure New composite Aging Cutting	W	_	al 10 days (1	Scotchbond Etchant 15 sec. Rinse 15 sec. Air 5 sec. Adhesive LC 10 sec. voling (5000X bit month speciments square test s	tw. 5 and 55 ° ens) and 11 m	onths storage	c. dwell time are (12 months sp	ecimens)	Clearfil Primer 20 sec. Air flow Bond LC 10 sec.	Scotchbond Etchant 15 sec. Rinse 15 sec. Air 5 sec. Adhesive LC 10 sec.	
Test specimen designation	Control	1a	1b	1c	2a	2b	2c	3a	3b	3с	
Number of test specimens (1 month)	45	41	41	45	43	44	64	42	58	75	
Number of test specimens (12 months)	44	52	40	40	41	61	58	57	59	53	

Each experimental group was further divided into subgroups, each receiving a different bonding system for repair: a—AdheSE One (Ivoclar Vivadent), a onestep self-etching adhesive; b—Clearfil SE Bond (Kuraray America Inc, New York, NY, USA), a two-step self-etching adhesive, and c—Adper Scotchbond Multi-Purpose (3M ESPE), a three-step etchand-rinse adhesive. All the adhesives were applied according to the manufacturers' recommendations for placement of composite restorations.

After surface treatment and adhesive application, the original mold was carefully placed over the cylinder and a second mold fitted on the top. The aged composite cylinders were then repaired using Tetric Evo Ceram, shade A2, in three incremental layers, the same way as the original specimens, resulting in 12-mm high specimens. After this, the cylinders were placed in distilled water for a week and then thermocycled 5000 times and finally stored in water for a total of 10 days. Control cylinders were also stored and thermocycled for same period of time. The eight cylinders in each test group were then divided into two groups to be tested either immediately (one month) or after a year of storage in distilled water (12 months). During the 12-month storage period, water was regularly replaced.

The composite cylinders were mounted on an automatic cutting machine (Isomet, Buehler Ltd, Lake Bluff, IL, USA) equipped with a thin, water-cooled diamond blade. The specimens were serially

sectioned perpendicularly to the bonding surface, both in the x-axis and the y-axis, producing a number of square test specimen rods approximately 1.1×1.1 mm. Ten to 20 test specimens were obtained from each composite cylinder. The test specimens were cleaned ultrasonically in distilled water for 3 minutes. After the cleaning procedure the test specimen rods were examined light microscopically at a magnification of $40\times$ for voids and imperfections in the composite and the adhesive interface and for evaluation of interface thickness. Only flawless specimen rods were tested. The width and thickness of each test specimen was measured to the nearest 0.01 mm using a calibrated digital caliper (Mitutoyo Co, Kawasaki, Japan).

A novel method was developed to attach the specimen rods to the bond testing machine to secure straight alignment of the rods and uniform distribution of the tensile forces throughout the specimens. The 1.1-mm size of the test specimen rods was selected to fit into the female (hollow) end of 2-mm commercially available male/female extension screws (ELRA AS, Oslo, Norway) (Figure 1). An extension screw was fitted to each end of the test specimen rods and secured with cyanoacrylate glue (Locktite 435, Henkel Norden, Gothenborg, Sweden). A custom-fit mold was made to ensure alignment of the screws to the long axis of the specimen.

Each test specimen was mounted in a calibrated universal testing machine (Lloyd Instruments Ltd,

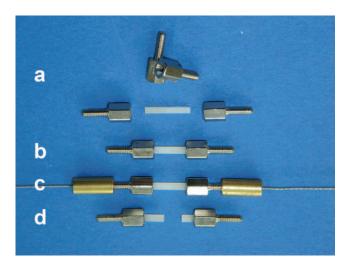


Figure 1. (a): The specimen rod to be tested and the 2-mm male/ female extension screws used. (b): The specimen rod fitted, aligned, and secured with cyanoacrylate glue. (c): The specimen assembly attached to the steel wires of the testing machine. (d): The specimen fractured at the repair joint.

Model LRX, Fareham, England) using specially attached steel wires designed to transmit pure tensile forces to the specimen. The microtensile testing was conducted at a crosshead speed of 1 mm/min until fracture. The tensile bond strength of each test specimen was calculated in megapascals by dividing the imposed force (in newtons) at fracture by the cross-sectional bond area (in millimeters squared). The test specimens were kept moist throughout the preparation of specimens and the test procedure.

The fracture surfaces were examined under a stereomicroscope (American Optical, Buffalo, NY, USA) at 40× magnification to determine whether the

failure region was within the adhesive zone or out of it. The adhesive zone was defined as the interface between the old and the new composite. The failure modes were classified as cohesive or adhesive.

The probability of failure in the test specimens was assessed by means of a distribution plot, and the significance of differences was evaluated by a mixed-model analysis of variance (ANOVA) and the Kolmogorov-Smirnov test. 58

RESULTS

The results are presented in Tables 3 to 5. The mean tensile strength of the unrepaired composite control group was 54.5 ± 6.0 MPa at one month and $49.6 \pm$ 5.1 MPa at 12 months. The lowest mean tensile strength in the repaired groups was for group 1c, mechanical roughening with sandpaper + Scotchbond Multi-Purpose, both at one month (26.4±6.8 MPa) and 12 months (21.2 \pm 9.9 MPa). This amounts to 48.4% of the strength of the control composite at one month and 42.7% of the strength at 12 months. The highest mean tensile strength for both storage periods was for group 3b, mechanical roughening + silane + Clearfil: 49.9 ± 10.4 at one month and 41.3 \pm 7.5 at 12 months, amounting to 91.6% and 83.3% of the control composite strength, respectively. There was a general reduction in the mean tensile strength for the repaired groups after 12 months of storage, amounting from 14.6% to 21.8% of the one-month tensile strength values. In general, Clearfil had the strongest repair strength within each surface treatment group. The mean tensile strength of the unrepaired control composite decreased 8.9% during the same storage period. Statistical calculations

Table 3: Results of Microtensile Testing in the Various Groups of Surface Treatment and Bonding Systems After One Month and 12 Months

Surface	Control	Mechanical Roughening (MR)				MR + CoJ	et	MR + Silane		
Treatment		1a AdheSE	1b Clearfil	1c Scotchbond	2a AdheSE	2b Clearfil	2c Scotchbond	3a AdheSE	3b Clearfil	3c Scotchbond
Bonding system										
Mean μTF (SD) (1 mo) ^a	54.5 (6.0)	28.6 (8.6)	40.2 (9.6)	26.4 (6.8)	40.5 (12.5)	45.4 (11.2)	35.6 (7.2)	43.2 (10.0)	49.9 (10.4)	35.2 (11.0)
Mean μTF (SD) (12 mo) ^a	49.6 (5.1)	24.1 (7.3)	33.6 (8.4)	21.2 (9.9)	32.9 (8.5)	36.8 (10.7)	30.4 (8.3)	33.8 (6.6)	41.3 (7.5)	28.2 (6.2)
Reduction in Mean μTF	8.9%	15.7%	16.4%	19.7%	18.8%	18.9 %	14.6%	21.8%	17.2%	19.9%
Mean μTF in % of contr. (1 mo) ^b	100%	52.5%	73.8%	48.4%	74.3%	83.3 %	65.3%	79.3%	91.6%	64.6%
Mean μTF in % of contr. (12 mo) ^b	100%	48.6%	67.7%	42.7%	66.3%	74.1 %	61.3%	68.1%	83.3%	56.9%

^a Mean microtensile force and standard deviation in MPa after one month (1 mo) and 12 months (12 mo).

^b Mean microtensile force in % of the mean value of control specimens after one month (1 mo) and 12 months (12 mo).

Surface Treatment	Control,	Mechanical Roughening (MR)			MR + CoJet			MR + Silane		
	%	1a AdheSE, %	1b Clearfil, %	1c Scotchbond, %	2a AdheSE, %	2b Clearfil, %	2c Scotchbond, %	3a AdheSE, %	3b Clearfil, %	3c Scotchbond %
Bonding system										
Cohesive fracture (1 mo)	100	4	2	2	0	7	0	5	16	4
Cohesive fracture (12 mo)	100	6	5	3	2	11	3	7	12	2

using ANOVA and Kolmogorov-Smirnov tests gave similar significance levels for all comparisons. At one month, there was a statistical difference between all groups, which was less pronounced at 12 months (Table 5). There was a tendency for lower tensile strength after one year compared with one month in all groups. However, the difference was not statistically significant. At both observation times, the mean strength of the control composite was significantly higher than that of the strongest repair.

The thickness of the bonding layer using Adper Scotchbond Multi-Purpose varied somewhat but appeared mostly to be approximately 175 μm ; the AdheSE One layer was approximately 20 μm ; and Clearfil SE, less than 5 μm .

The percentage of cohesive fractures for each group is presented in Table 4. All the cohesive fractures occurred in the old composite. Most cohesive fractures for the repair groups, 16% of specimens at one month and 12% at 12 months, were in group 3b, which also had the highest mean repair strength for both storage periods; group 2b had 7% at 1 month and 11% at 12 months and came in second

in mean tensile repair strength. Other groups had fewer or no cohesive failures.

DISCUSSION

Shear bond strength tests have been widely used when measuring adhesion to dental structures or dental materials. The wide acceptance of this testing method is due to its relative simplicity when compared with tensile strength methods. Some authors have found that conventional shear bond testing produced stress concentrations in the substrate or adherend, leading to cohesive failures when testing adhesive joints and therefore giving misleading results.^{59,60} This has led to increased use of the microtensile test where, in a very small specimen, comparatively more uniform loading stress distribution is obtained and the tensile forces concentrated in the adhesive interface are tested. 61,62 In a review on microtensile bond testing, Pashlev⁶² stated that microtensile testing offered versatility not obtainable with other methods even though it is more labor intensive. Poitevin and others⁶³ reported on composite adhesive strength to dentin and concluded that microtensile testing was a reliable laboratory test in

Table 5:	Results of Sta Between Gro			alysis of Varia Months*	ance and Koln	nogorov-Smirr	ov Tests Eval	uating the Di	fference
Groups	1a	1b	1c	2a	2b	2c	3a	3b	3c
control	A, b	A, b	A, b	A, b	A, b	A, b	A, b	A, b	A, b
1a		A, b	A, ns	A, b	A, b	A, b	A, b	A, b	A, b
1b			A, b	A, ns	A, ns	A, ns	A, ns	A, b	A, b
1c				A, b	A, b	A, b	A, b	A, b	A, b
2a					A, ns	A, ns	A, ns	A, b	A, ns
2b						A, b	A, ns	A, b	A, b
2c							A, ns	A, b	A, ns
3a								A, b	A, b
3b									A, b
3c									

Abbreviation: ns, difference between groups not statistically significant.

^{*} Uppercase letter represents one month and lowercase letter represents 12 months storage. Similar letters represent statistical difference (p<0.05).

ranking contemporary adhesives on their bonding effectiveness. Recently, a microshear test was introduced to measure bond strength between dentin and resin composite, intended as an alternative to microtensile testing. The main reason given for using the microshear test was it required a less demanding and time-consuming specimen collection. Only one investigation on composite repair using microshear bond testing was found.

Several authors⁶⁶ have found some evidence for correlation of laboratory bond strength with clinical retention rates of class V restorations. In a recent study, Heintze⁶⁷ found that both macrotensile and microtensile bond strength tests correlated better with clinical retention of cervical restorations than macroshear and microshear testing. He recommended shear testing to be abandoned due to critical and inadequate stress distribution and unreliable correlation to clinical outcome. Because no universal agreement exists on bonding methods, these authors decided after preliminary investigation to use microtensile strength testing.

For this investigation an easier and much less time-consuming method was developed to measure the tensile bond strength, where straight alignment of the specimen rods glued into commercially available and inexpensive extension screws was obtained. The specimen/extension screw assembly, screwed to the aligned steel wires attached to the testing machine, directed the pulling force longitudinally from the ends of the specimen. Under these testing conditions a vast majority of specimen rods broke in the repair junction. In preliminary testing of the methods and materials used in this study, one flat side of the specimen was glued directly to the jig of the testing machine, resulting in cohesive fractures much more often than when the extensionscrew setup was tested. This was attributed to the possibility that the forces might have been less uniformly distributed throughout the specimen than in the new method.⁶⁸ In all the other comparable investigations ^{19,22,25,43,69} on repair, where microtensile testing was used, cohesive failure is reported as very high, up to 95%. The number of cohesive fractures reported in some of these studies is surprising, because it could be anticipated that the adhesive joint would be the weakest link. If the cohesive fractures are relatively few, as in this study, the results more likely demonstrate the true repair strength of the adhesive joint between the old and the new composites. In general, more cohesive fractures can be expected as the repair strength approaches the fracture strength of the composite used, as observed in this study. However, when the repair strength measured is only half the cohesive strength of the composite used, and two-thirds of the fractures are reported as cohesive, the testing procedure must be questioned.¹⁹

When the three bonding systems tested in this study are compared separately within each surface treatment group, it is obvious that Adper Scotchbond Multi-Purpose, the three-step adhesive, gave the lowest repair microtensile bond strength.

The three-step adhesives have been classified as the criterion standard in adhesive technology when placing resin composite restorations, especially large posterior restorations in load-bearing areas. 70-72 The great success this group of materials has experienced is possibly because the relatively thick and more flexible adhesive layer serves as a shock absorber between tooth and composite when the restoration is under masticatory stresses. 53,56 Another explanation could be that it contains less hydrophilic monomers than the more acidic self-etching adhesives. High hydrophilicity of an adhesive system may impair the long-term durability of the adhesive, because hydrophilic monomers tend to absorb more water, in time weakening the bond. 16,26 One study 43 on repair of composites reported early signs of degradation for hydrophilic self-etching adhesive repair bond compared with a hydrophobic three-step adhesive at six months. In most studies 24-48 the specimens are tested immediately or a few weeks after the composite repair, and no studies 23,43 could be found with storage periods exceeding six months. In this study the specimens were tested after one and 12 months of storage in water in addition to thermocycling. Between the testing periods, a general reduction in mean tensile strength values, 16% to 22%, was observed for all the repaired groups (Table 4). These differences, however, were not statistically significant. A slight reduction, 8.9%, was also observed for the control composite specimens between the storage periods.

When gluing hard pieces together it is of utmost importance to wet both surfaces with the appropriate glue and bring the pieces as tightly together as possible. Evaluating the results and mode of failure from this study, it is postulated that the adhesive film thickness plays a role when repairing resin composite with resin composite. The components in the Clearfil system are considerably more fluid than those in the AdheSE One and the Adper Scotchbond Multi-Purpose adhesives, therefore producing a thinner adhesive layer. Almost all the Adper Scotchbond Multi-Purpose and the AdheSE One specimens failed

in the adhesive layer, and many specimens showed adhesive material on both the repaired and the new composite, indicating that the tensile strength of the adhesive itself directed the repair tensile strength at failure. In the Clearfil specimens, the fracture appeared to occur more between the old composite and the adhesive, except for group 3b, the silane-Clearfil group that showed the strongest mean repair bond, which had almost 92% of the mean cohesive strength of the control composite at one month and more than 83% at 12 months. There the fracture often appeared both between the old and the new composite and the adhesive in the same specimen. Coelho and others⁵⁶ observed lower microtensile bond strength values for Single Bond (3M ESPE) as the adhesive layer between dentin and composite was increased from one layer to three layers; whereas, no correlation could be found for the more fluid Clearfil SE (Kuraray). In that investigation, however, the majority of specimens for both adhesive systems presented mixed cohesive/adhesive failure mode.⁵⁶

In this investigation three surface treatments of the aged composite were used. The purpose of a surface treatment is to increase the surface energy and/or the surface roughness. A common practice for mechanical roughening of composite specimens is to use silicon carbide sandpaper with a specific grit, giving a standardized surface roughening. Use of 320-grit sandpaper has been selected by several investigators, 23,69,73 the same grit selected for this study. Both of the other two surface treatments added sandblasting the surface with CoJet silica-coated alumina particles or silane application to the 320-grit sandpaper roughening, which significantly improved the mean bond strength (p<0.05), except for group 2b at 12 months. The CoJet particles are designed to penetrate and be embedded in the surface of the substrate and leave it partially coated with silica.⁷⁴ It is possible that the embedded particles act as microretention for the new composite, explaining the improved bond strength. Sandblasting using alumina particles has also been reported to improve repair bond strength. 25,43 In one study²² no difference was found between sandblasting with aluminum oxide and silica coating with CoJet. It was postulated that similar surface roughness pattern resulted in similar mechanical retention.

It is a known fact that silane bonds well to silicabased materials and also to resin composites. Many resin composites contain filler materials that consist of siliceous compounds. This can explain why silanization of old composite improves bonding to new composite as demonstrated in this study. Some studies^{22,28,43} are, however, in disagreement with our findings. One-bottle prehydrolyzed silane solutions have a relatively short shelf life and gradually become less reactive after opening of the bottle, preventing optimal adhesion. In this study a freshly mixed (two-bottle) silane system was used. The two solutions are mixed just before use to allow hydrolysis of the silane to secure a fresh reactive solution. Lundvall and others found a much higher bond strength when repairing porcelain with composite using two-bottle silane, whereas one-bottle silane showed similar bond strength as the group without silane.

Using air abrasion in the mouth requires an intraoral sandblaster and it can be difficult to achieve in the dental operatory. Using silane as an adhesion promoter is a simple method that requires no extra equipment and, according to this study, achieves comparable or better results. Within the limitations of this study, a two-bottle silane adhesion promoter in addition to a thin layer of adhesive would be the treatment of choice when repairing resin composite.

CONCLUSIONS

The results obtained did not support the three null hypotheses that were claimed. The best repair bond was achieved by using freshly mixed silane solution in addition to an adhesive rendering a thin bonding layer.

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Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES

- Mjor IA (1997) The reasons for replacement and the age of failed restorations in general dental practice Acta Odontologica Scandinavica 55(1) 58-63.
- 2. Mjor IA, & Gordan VV (2002) Failure, repair, refurbishing and longevity of restorations *Operative Dentistry* **27(5)** 528-534.
- 3. Mjor IA, Shen C, Eliasson ST, & Richter S (2002) Placement and replacement of restorations in general dental practice in Iceland *Operative Dentistry* **27(2)** 117-123.

 Tyas MJ, Anusavice KJ, Frencken JE, & Mount GJ (2000) Minimal intervention dentistry—A review FDI Commission Project 1-97 International Dental Journal 50(1) 1-12.

- Krejci I, Lieber CM, & Lutz F (1995) Time required to remove totally bonded tooth-colored posterior restorations and related tooth substance loss. *Dental Materials* 11(1) 34-40.
- Gordan VV, Riley JL, Worley DC, & Gilbert GH (2012) Restorative material and other tooth-specific variables associated with the decision to repair or replace defective restorations: Findings from The Dental PBRN Journal of Dentistry 40(5) 397-405.
- Mjor IA (1993) Repair versus replacement of failed restorations International Dental Journal 43(5) 466-472.
- Blum IR, Lynch CD, Schriver A, Heidemann D, & Wilson NH (2011) Repair versus replacement of defective composite restorations in dental schools in Germany European Journal of Prosthodontics and Restorative Dentistry 19(2) 56-61.
- Lynch CD, Blum IR, Frazier KB, Haisch LD, & Wilson NH (2012) Repair or replacement of defective direct resinbased composite restorations: Contemporary teaching in U.S. and Canadian dental schools *Journal of the Amer*ican Dental Association 143(2) 157-163.
- Blum IR, Lynch CD, & Wilson NH (2012) Teaching of the repair of defective composite restorations in Scandinavian dental schools *Journal of Oral Rehabilitation* 39(3) 210-216.
- Blum IR, Lynch CD, & Wilson NH (2012) Teaching of direct composite restoration repair in undergraduate dental schools in the United Kingdom and Ireland European Journal of Dental Education 16(1) e53-e58.
- Gordan VV, Mjor IA, Blum IR, & Wilson NH (2003)
 Teaching students the repair of resin-based composite restorations: A survey of North American dental schools Journal of the American Dental Association 134(3) 317-323, quiz 338-339.
- Gordan VV, Riley JL, Blaser PK, & Mjor IA (2006) 2-year clinical evaluation of alternative treatments to replacement of defective amalgam restorations *Operative Den*tistry 31(4) 418-425.
- Boyer DB, Chan KC, & Torney DL (1978) The strength of multilayer and repaired composite resin *Journal of Prosthetic Dentistry* 39(1) 63-67.
- Brosh T, Baharav H, Gross O, & Laufer BZ (1997) The influence of surface loading and irradiation time during curing on mechanical properties of a composite *Journal of Prosthetic Dentistry* 77(6) 573-577.
- Malacarne J, Carvalho RM, de Goes MF, Svizero N, Pashley DH, Tay FR, Yiu CK, & Carrilho MR (2006) Water sorption/solubility of dental adhesive resins *Dental Materials* 22(10) 973-980.
- Lagouvardos PE, Pissis P, Kyritsis A, & Daoukaki D (2003) Water sorption and water-induced molecular mobility in dental composite resins *Journal of Materials* Science: Materials in Medicine 14(9) 753-759.
- 18. Tarumi H, Torii M, & Tsuchitani Y (1995) Relationship between particle size of barium glass filler and water

- sorption of light-cured composite resin *Dental Materials Journal* **14(1)** 37-44.
- Maneenut C, Sakoolnamarka R, & Tyas MJ (2011) The repair potential of resin composite materials *Dental* Materials 27(2) e20-e27.
- da Costa TR, Serrano AM, Atman AP, Loguercio AD, & Reis A (2012) Durability of composite repair using different surface treatments *Journal of Dentistry* 40(6) 513-521.
- Dall'oca S, Papacchini F, Radovic I, Polimeni A, & Ferrari M (2008) Repair potential of a laboratory-processed nanohybrid resin composite *Journal of Oral Science* 50(4) 403-412.
- Rodrigues SA Jr, Ferracane JL, & Della Bona A (2009)
 Influence of surface treatments on the bond strength of repaired resin composite restorative materials *Dental Materials* 25(4) 442-451.
- Staxrud F, & Dahl JE (2011) Role of bonding agents in the repair of composite resin restorations *European Journal* of Oral Science 119(4) 316-322.
- 24. Costa TR, Ferreira SQ, Klein-Junior CA, Loguercio AD, & Reis A (2010) Durability of surface treatments and intermediate agents used for repair of a polished composite Operative Dentistry 35(2) 231-237.
- Loomans BA, Cardoso MV, Roeters FJ, Opdam NJ, De Munch J, Huysmans MC, & Van Meerbeek B (2011) Is there one optimal repair technique for all composites? Dental Materials 27(7) 701-709.
- Papacchini F, Toledano M, Monticelli F, Osorio R, Radovic I, Polimeni A, Garcia-Godoy F, & Ferrari M (2007) Hydrolytic stability of composite repair bond European Journal of Oral Science 115(5) 417-424.
- 27. Passos SP, Ozcan M, Vanderlei AD, Leite FP, Kimpara ET, & Bottino MA (2007) Bond strength durability of direct and indirect composite systems following surface conditioning for repair *Journal of Adhesive Dentistry* 9(5) 443-447.
- Bonstein T, Garlapo D, Donarummo J Jr, & Bush PJ (2005) Evaluation of varied repair protocols applied to aged composite resin *Journal of Adhesive Dentistry* 7(1) 41-49.
- 29. Cavalcanti AN, De Lima AF, Peris AR, Mitsui FH, & Marchi GM (2007) Effect of surface treatments and bonding agents on the bond strength of repaired composites Journal of Esthetic and Restorative Dentistry 19(2) 90-98, discussion 99.
- Rathke A, Tymina Y, & Haller B (2009) Effect of different surface treatments on the composite-composite repair bond strength Clinical Oral Investigations 13(3) 317-323.
- 31. Yesilyurt C, Kusgoz A, Bayram M, & Ulker M (2009) Initial repair bond strength of a nano-filled hybrid resin: Effect of surface treatments and bonding agents *Journal of Esthetic and Restorative Dentistry* **21(4)** 251-260.
- Padipatvuthikul P, & Mair LH (2007) Bonding of composite to water aged composite with surface treatments *Dental Materials* 23(4) 519-525.
- 33. Papacchini F, Dall'Oca S, Chieffi N, Goracci C, Sadek FT, Suh BI, Tay FR, & Ferrari M (2007) Composite-tocomposite microtensile bond strength in the repair of a

- microfilled hybrid resin: Effect of surface treatment and oxygen inhibition *Journal of Adhesive Dentistry* **9(1)** 25-31.
- 34. Papacchini F, Magni E, Radovic I, Mazzitelli C, Monticellia F, Goracci C, Polimeni A, & Ferrari M (2007) Effect of intermediate agents and pre-heating of repairing resin on composite-repair bonds *Operative Dentistry* **32(4)** 363-371
- 35. Papacchini F, Monticelli F, Radovic I, Chieffi N, Goracci C, Tay FR, Polimeni A, & Ferrari M (2007) The application of hydrogen peroxide in composite repair Journal of Biomedical Materials Research Part B: Applied Biomaterials 82(2) 298-304.
- Papacchini F, Radovic I, Magni E, Goracci C, Monticelli F, Chieffi N, Polimeni A, & Ferrari M (2008) Flowable composites as intermediate agents without adhesive application in resin composite repair American Journal of Dentistry 21(1) 53-58.
- 37. Kupiec KA, & Barkmeier WW (1996) Laboratory evaluation of surface treatments for composite repair *Operative Dentistry* **21(2)** 59-62.
- Shahdad SA, & Kennedy JG (1998) Bond strength of repaired anterior composite resins: an in vitro study Journal of Dentistry 26(8) 685-694.
- 39. Yap AU, Quek CE, & Kau CH (1998) Repair of newgeneration tooth-colored restoratives: Methods of surface conditioning to achieve bonding *Operative Dentistry* **23(4)** 173-178.
- 40. Yap AU, Sau CW, & Lye KW (1999) Effects of aging on repair bond strengths of a polyacid-modified composite resin *Operative Dentistry* **24(6)** 371-376.
- Lucena-Martin C, Gonzalez-Lopez S, & Navajas-Rodriguez de Mondelo JM (2001) The effect of various surface treatments and bonding agents on the repaired strength of heat-treated composites *Journal of Prosthetic Dentistry* 86(5) 481-488.
- 42. Oztas N, Alacam A, & Bardakcy Y (2003) The effect of air abrasion with two new bonding agents on composite repair *Operative Dentistry* **28(2)** 149-154.
- 43. da Costa TRF, Serrano AM, Atman AP, Loguercio AD, & Reis A (2012) Durability of composite repair using different surface treatments *Journal of Dentistry* 40(6) 513-521.
- Loomans BAC, Cardoso MV, Opdam NJ, Roeters FJ, De Munck J, Huismans MC, & Van Meerbeek B (2011) Surface roughness of etched composite resin in light of composite repair *Journal of Dentistry* 39(7) 499-505.
- Trajtenberg CP, & Powers JM (2004) Effect of hydrofluoric acid on repair bond strength of a laboratory composite American Journal of Dentistry 17(3) 173-176.
- 46. Brendeke J, & Ozcan M (2007) Effect of physicochemical aging conditions on the composite-composite repair bond strength *Journal of Adhesive Dentistry* **9(4)** 399-406.
- 47. Fawzy AS, El-Askary FS, & Amer MA (2008) Effect of surface treatments on the tensile bond strength of repaired water-aged anterior restorative micro-fine hybrid resin composite *Journal of Dentistry* 36(12) 969-976.
- 48. Özcan M, Barbosa SH, Melo RM, Galhano GA, & Bottino MA (2007) Effect of surface conditioning methods on the

- microtensile bond strength of resin composite to composite after aging conditions *Dental Materials* **23(10)** 1276-1282.
- 49. Hamano N, Chiang YC, Nyamaa I, Yamaguchi H, Ino S, Hickel R, & Kunzelmann KH (2012) Repair of silorane-based dental composites: Influence of surface treatments *Dental Materials* **28(8)** 894-902.
- 50. Wiegand A, Stawarczyk B, Buchalla W, Taubock TT, Ozcan M, & Attin T (2012) Repair of silorane composite—
 Using the same substrate or a methacrylate-based composite? *Dental Materials* **28(3)** e19-e25.
- 51. Ivanovas S, Hickel R, & Ilie N (2011) How to repair fillings made by silorane-based composites *Clinical Oral Investigations* **15(6)** 915-922.
- 52. Giachetti L, Russo DS, Baldini M, Goracci C, & Ferrari M (2012) Reparability of aged silorane with methacrylatebased resin composite: Micro-shear bond strength and scanning electron microscopy evaluation *Operative Den*tistry 37(1) 28-36.
- Kemp-Scholte CM, & Davidson CL (1990) Complete marginal seal of class V resin composite restorations effected by increased flexibility *Journal of Dental Re*search 69(6) 1240-1243.
- Knight GT, & Berry TG (1997) Clinical application of a direct placement mercury-free alloy American Journal of Dentistry 10(1) 52-54.
- Tam LE, Khoshand S, & Pilliar RM (2001) Fracture resistance of dentin-composite interfaces using different adhesive resin layers *Journal of Dentistry* 29(3) 217-225.
- 56. Coelho PG, Calamia C, Harsono M, Thompson VP, & Silva NR (2008) Laboratory and FEA evaluation of dentin-to-composite bonding as a function adhesive layer thickness *Dental Materials* 24(10) 1297-1303.
- 57. Kalachandra S (1989) Influence of fillers on the water sorption of composites *Dental Materials* **5(4)** 283-288.
- Press WP, Flannery BP, Teukolsky SA, & Vetterling WT (1986) Kolmogorov-Smirnov test In: Numerical Recipes: The Art of Scientific Computing Cambridge University Press, Cambridge 472-475.
- Van Noort R, Noroozi S, Howard IC, & Cardew G (1989) A critique of bond strength measurements *Journal of Dentistry* 17(2) 61-67.
- Della Bona A, & van Noort R (1995) Shear vs. tensile bond strength of resin composite bonded to ceramic *Journal of Dental Research* 74(9) 1591-1596.
- 61. Scherrer SS, Cesar PF, & Swain MV (2010) Direct comparison of the bond strength results of the different test methods: A critical literature review *Dental Materials* **26(2)** E78-E93.
- 62. Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA, & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1(4)** 299-309.
- 63. Poitevin A, De Munck J, Van Landuyt K, Coutinho E, Peumans M, Lambrechts P, & Van Meerbeek B (2008) Critical analysis of the influence of different parameters on the microtensile bond strength of adhesives to dentin *Journal of Adhesive Dentistry* **10(1)** 7-16.

64. McDonough WG, Antonucci JM, He J, Shimada Y, Chiang MY, Schumacher GE, & Schultheisz CR (2002) A microshear test to measure bond strengths of dentin-polymer interfaces *Biomaterials* **23(17)** 3603-3608.

- 65. Foong J, Lee K, Nguyen C, Tang G, Austin D, Ch'ng C, Burrow MF, & Thomas DL (2006) Comparison of microshear bond strengths of four self-etching bonding systems to enamel using two test methods *Australian Dental Journal* **51(3)** 252-257.
- 66. Van Meerbeek B, Peumans M, Poitevin A, Mine A, Van Ende A, Neves A, & De Munck J (2010) Relationship between bond-strength tests and clinical outcomes *Dental Materials* 26(2) E100-E121.
- 67. Heintze SD (2013) Clinical relevance of tests on bond strength, microleakage and marginal adaptation *Dental Materials* **29(1)** 59-84.
- 68. El Zohairy AA, de Gee AJ, de Jager N, van Ruijven LJ, & Feilzer AJ (2004) The influence of specimen attachment and dimension on microtensile strength *Journal of Dental Research* 83(5) 420-424.
- 69. Hamano N, Chiang YC, Nyamaa I, Yamaguchi H, Ino S, Hickel R, & Kunzelmann KH (2012) Repair of silorane-

- based dental composites: Influence of surface treatments *Dental Materials* **28(8)** 894-902.
- Van Meerbeek B, De Munck L, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, & Vanherle G (2003) Buonocore Memorial Lecture: Adhesion to enamel and dentin: Current status and future challenges Operative Dentistry 28(3) 215-235.
- Perdigao J (2007) New developments in dental adhesion Dental Clinics of North America 51(2) 333-357, viii.
- Manuja N, Nagpal R, & Pandit IK (2012) Dental adhesion: Mechanism, techniques and durability Journal of Clinical Pediatric Dentistry 36(3) 223-234.
- 73. Tezvergil A, Lassila LVJ, & Vallittu PK (2003) Composite-composite repair bond strength: Effect of different adhesion primers *Journal of Dentistry* **31(8)** 521-525.
- Lung CYK, & Matinlinna JP (2012) Aspects of silane coupling agents and surface conditioning in dentistry: An overview. *Dental Materials* 28(5) 467-477.
- Lundvall PK, Ruyter E, Ronold HJ, & Ekstrand K (2009)
 Comparison of different etching agents and repair materials used on feldspathic porcelain *Journal of Adhesion Science and Technology* 23(7-8) 1177-1186.