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Article *in* Journal of Conservative Dentistry · May 2018

DOI: 10.4103/JCD.JCD_11_18

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ISSN: 0972-0707

Journal of Conservative Dentistry

Volume 21 Issue 3 May-June 2018

Official Publication of
Indian Association of Conservative Dentistry and Endodontics

Online full text at
www.jcd.org.in

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Bonding performance of universal adhesives on composite repairs, with or without silane application

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Abstract

Aim: This study aims to investigate the adhesive performance of three different universal adhesives to repair aged composite restorations, with or without the application of silane.

Materials and Methods: A hundred and twenty resin composite samples were prepared, aged and randomly divided into 6 main Groups (single bond universal [SBU], All-Bond Universal [ABU], Futurabond U, Clearfil Tri-S Bond, Single Bond 2, and Clearfil SE Bond) and 2 subgroups (with or without silane). A microhybrid composite resin was placed on the aged composite surfaces and light cured. After a micro-shear bonding test, the fracture surfaces were examined under the scanning electron microscopy. Statistical analysis was performed using two-way ANOVA and Tukey's *post hoc* tests.

Results: Among all the universal adhesives, SBU showed the highest bond strength values compared to the other two universal adhesives when used with and without silane ($P > 0.05$). Between ABU and Futurabond U, no significant difference was observed with silane ($P > 0.05$) and without silane ($P > 0.05$). Among conventional adhesives, there was no statistically significant difference ($P > 0.05$) both with and without silane.

Conclusion: Within the limitations of this study, while SBU can be safely used with and without the application of silane, Futurabond U cannot be used without silane.

Keywords: Composite repair; micro-shear bond strength; silane application; universal adhesives

INTRODUCTION

The replacement of defective restorations is sometimes required in general dental practice due to secondary caries, marginal defects, aged restorations or cuspal fracture, and insufficient marginal integrity.[1,2] With the repairing of defective areas instead of replacement, it is possible to protect both the dental tissues and restorative material because less hard tissue is removed.[3] The repairing of an aged restoration causes less destruction compared to the replacement of the restoration, with a reduced risk of tooth fracture and pulp damage.[4] Previous studies have shown that alternative treatments to the replacement of defective restorations, such as marginal sealing, and refurbishment for defective restorations and repair, have significantly improved the clinical properties and increased their quality and longevity with minimal intervention.[5,6]

Researchers have reported that additional surface pretreatment procedures were necessary to obtain adequate bond strength with regard to resin materials used in aged composite restorations.[7,8] They have suggested the possibility of surface treatment of the original composite (OC), including mechanical roughening, acid "etching," the application of low viscosity bonding agents, "flowable" composites, and silane.[9-12] Maneenut *et al*. reported that silane-based adhesives had stronger bond strength than that of adhesives without silane.[7]

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Date of submission : 16.02.2018
Review completed : 26.01.2018
Date of acceptance : 21.02.2018

Access this article online
Quick Response Code:
Website: www.jcd.org.in
DOI: 10.4103/JCD.JCD_11_18

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How to cite this article: Çakir NN, Demirbuga S, Balkaya H, Karadaş M. Bonding performance of universal adhesives on composite repairs, with or without silane application. J Conserv Dent 2018;21:263-8.

Clinicians have to keep a range of different materials in their clinics for use in repairing aged composite restorations to ensure adequate bonding strength. This can be expensive. Furthermore, additional application steps are time-consuming for both clinicians and patients. Therefore, manufacturers have introduced multipurpose “universal one-bottle adhesives” for use as either self-etch or etch and rinse adhesives. Perdigao and Loguercio also claimed that these adhesives can be bonded to all kinds of hard tissues or materials.[13]

Universal adhesives have similar composition to conventional one-step self-etch adhesives. Most of the adhesives contain specific carboxylate and/or phosphate monomers which bond chemically to calcium in hydroxyapatite.[14] In addition, universal adhesives contain both water and at least, one organic solvent like ethanol or acetone. Universal adhesives are easy to use and provide faster application procedure and less technique-sensitive to clinicians when compared with multi-step adhesives.[15]

There is a lack of literature with regard to information on the use of universal adhesives in the repair of composite restorations. For this reason, the aim of this study was to evaluate the adhesive performance of universal adhesives when composite resins were used to repair aged composite restorations. The null hypothesis of the present study was that there is no difference between universal adhesives when used with or without silane with regard to the repair of aged composite restorations.

MATERIALS AND METHODS

The brand name, manufacturer, chemical composition, and batch number of the materials used in the current study are listed in Table 1.

Preparation of composite specimens

A total of 120 composite specimens (Charisma, Heraeus Kulzer) (6 mm diameter × 2 mm thickness) were prepared in plastic molds. The mold was positioned on a glass microscope slide, and the composite resin was injected, a second microscope slide was pressed firmly onto the composite surface to remove the excess, and then light-cured for 20 s using a blue light emitting diode device (LED) (Elipar Free Light III, 3M ESPE®, St. Paul, MN, USA) at 1200 mW/cm^2 output. The intensity of the light-curing unit was controlled throughout the experiment using a radiometer (Demetron LED Radiometer, Kerr Sybron Dental Specialties, Middleton, WI, USA). The samples were polished with a series of aluminum oxide polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA) under constant water cooling. After polymerization, the samples were first water-stored at 37°C for 48 h, boiled for 8 h in water, and again water-stored at 37°C for 3 weeks according to the aging method described previously.[16] The surface of all samples was sandblasted with 50 µm Al_2O_3 (KaVo RONDOflex 2015 Powder; KaVo Dental GmbH, Biberach, Germany) using an intraoral sandblaster (KaVo RONDOflex Plus 360; KaVo Dental GmbH) at a pressure of 2 bars, from a distance of 10 mm for 10 s. For one-half of the groups, silane (Clearfil Ceramic Primer, Kuraray Medical, Inc., Okayama, Japan) was applied to the surface of the aged composites according to the manufacturer’s instructions [Table 1].

Experimental groups and adhesive applications

The composite specimens were divided into 12 Groups according to the adhesive applications as mentioned below:

- Group 1: Single bond universal (SBU) (3M ESPE)
- Group 2: Silane (Kuraray) + SBU (3M ESPE)
- Group 3: Futurabond U (Voco)
- Group 4: Silane (Kuraray) + Futurabond U (Voco)
- Group 5: All-Bond Universal (ABU) (Bisco)
- Group 6: Silane (Kuraray) + ABU (Bisco)
- Group 7: Single bond 2 (SB2) (3M ESPE)
- Group 8: Silane (Kuraray) + SB2 (3M ESPE)
- Group 9: Clearfil Tri-S Bond (TSB) (Kuraray)
- Group 10: Silane (Kuraray) + Clearfil TSB (Kuraray)
- Group 11: Clearfil SE bond (CSE) (Kuraray)
- Group 12: Silane (Kuraray) + CSE (Kuraray).

All adhesive procedures were performed at room temperature (24°C) and 70% relative humidity according to manufacturer’s instructions as mentioned in Table 1.

Preparation of composite cylinders

Three cylindrical composite buildups (Charisma, Heraeus Kulzer) were applied to each composite surface (n = 20 on 10 composite discs, placing 2 composite cylinders on each composite disc) using Tygon tubes (0.75 mm internal diameter × 1 mm length) (Tygon, Norton Performance Plastic Co, Cleveland, OH, USA) and bulk-cured for 20 s.

Micro-shear bond strength test

After storage in distilled water (37°C/24 h), the Tygon tubes were removed using a scalpel and the specimens were subjected to a universal testing machine (Instron, Model 4444, Instron Corporation, Canton, MA, USA). A 0.25 mm thick wire loop was placed around the composite cylinders in contact with semi-peripherally. A micro-shear bond test was performed at a crosshead speed of 0.5 mm/min. The micro-shear forces were recorded in Newtons (N) and calculated as megapascals (MPa) dividing by the bonding area (mm^2).

Scanning electron microscopy analysis of debonded surfaces

All fracture surfaces of the debonded samples were coated with gold pallium and examined using scanning electron microscopy (SEM) (LEO-440, Zeiss, Cambridge,

Table 1: Materials, composition, and application modes used in present study

Materials	Composition	Application mode
Charisma Heraeus Kulzer, Hanau, Germany Batch# 010417A	Bis-GMA, TEGDMA, barium aluminum fluoride glass, silicon dioxide	Apply resin composite to surface Light polymerize for 20 s
Clearfil SE Bond Kuraray, Osaka, Japan Primer Batch# 01041A Bond Batch# 01552A	Primer: MDP, HEMA, hydrophilic dimethacrylate, dl-camphorquinone, N, N-diethanol-p-toluidine, water. Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, dl-camphorquinone, N, N-diethanol-p-toluidine, silanated colloidal silica	Apply primer to tooth surface and leave in place for 20 s Dry with air stream to evaporate the volatile ingredients Apply bond to the tooth surface and then create a uniform film using a gentle air stream Light polymerize for 10 s
Adper Single Bond 2 3M ESPE, St. Paul, MN, USA Batch# N151635	HEMA, Bis-GMA, ethanol, dimethacrylate, methacrylate functional copolymer of polyacrylic and polytaconic acid, water, photoinitiator	Apply etchant for 15 s Rinse for 10 s Blot excess water Apply 2-3 consecutive coats of adhesive for 15 s with gentle agitation Gently air dry for 5 s Light polymerize for 10 s
CLEARFIL TRI-S BOND Kuraray Medical Inc., Okayama, Japan Batch# 000004	MDP, Bis-GMA, HEMA, colloidal silica, ethanol, water, dl-camphorquinone, initiators, accelerators, others	Apply adhesive for 20 s Air dry for more than 5 s Light polymerize for 10 s
Futurabond U Voco, Cuxhaven, Germany Batch# 1415274	Liquid 1: acidic adhesive monomer, HEMA, BISGMA, HEDMA, UDMA, catalyst. liquid 2: Ethanol, initiator, catalyst	Mix and stir thoroughly both liquids with the Single Tim applicator Apply the adhesive homogenously to the surface and rub for 20 s using the single Tim Dry off the adhesive layer with dry, oil-free air for at least 5 s Light cure the adhesive layer for 10 s
Single Bond Universal Adhesive 3M ESPE, Neuss, Germany Batch# 535812	10-MDP phosphate monomer, Vitrebond, copolymer, HEMA, BISGMA, dimethacrylate resins, filler, silane, initiators, ethanol, water	Apply the adhesive with the applicator to the entire surface and rub for 20 s Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely Harden the adhesive with a curing light for 10 s
All-Bond Universal Bisco, Schaumburg, USA Batch# 1400007671	10-MDP phosphate monomer, HEMA, BISGMA, ethanol, water, initiators	Dispense 1-2 drops of ABU into a clean well Apply two separate coats, scrubbing the preparation with a microbrush for 10-15 s per coat Evaporate excess solvent by thoroughly air-drying for at least 10 s. Surface should have a uniform glossy appearance Light cure for 10 s
Clearfil Silane Kuraray Noritake Dental Inc., Okayama, Japan Batch# 440030	6-(4-vinylbenzyl-n-propyl) amino-1,3,5 triazine-2,4-dithiol,-dithione tautomer and 10-MDP in acetone	Apply silane coupling agent and wait for 5 s Dry with oil-free air

Bis-GMA: Bisphenol A diglycidyl methacrylate, HEMA: 2-hydroxyethyl methacrylate, TEGDMA: Triethylene glycol dimethacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, UDMA: Urethane dimethacrylate, HEDMA: 1,6-hexanediol dimethacrylate, Al_2O_3: Aluminum oxide, ABU:

England). The failure modes were classified as follows: Adhesive failure (A); at repair composite (RC)-OC interface, cohesive failure in RC; fracture in RC, cohesive failure in OC; fracture in OC, mixed failure (M); a combination of adhesive/cohesive failure.

Statistical analysis

Statistical analysis was undertaken using SPSS 10.0 software (SPSS Inc., Chicago, IL, USA). For each group (Current vs. Universal and silane vs. nonsilane), bond strength data were analyzed using a two-way ANOVA to detect any statistical differences, and multiple comparisons were done using Tukey's *post hoc* test. Fracture modes were analyzed using the Chi-square test. Pretesting failures were not included in the statistical analysis.

RESULTS

Table 2 shows the mean micro-shear bond strength values of the groups. Among all the silane groups, SBU provided the highest bond strength (49.3 ± 13.7 MPa) followed by CSE (44.4 ± 12.1 MPa) and SB2 (40.2 ± 11.9 MPa), respectively. SBU showed the highest bond strength values compared to the other two universal adhesives when used with and without silane ($P > 0.05$). Between ABU and Futurabond U no significant difference was observed with ($P > 0.05$) and without silane ($P > 0.05$).

Among conventional adhesives, there was no statistically significant difference ($P > 0.05$) both with and without silane. Only the bond strength of Futurabond U increased when used with silane compared to nonsilane groups (21.5 ± 8.1–36.9 ± 11.2 MPa) ($P < 0.05$).

In groups without the use of silane, SBU showed the highest bond strength (47.4 ± 13.0 MPa) followed by SB2 (39.2 ± 11.3 MPa). When SBU was applied without silane, it provided similar bond strength with two conventional adhesives, SB2 and CSE when used with silane ($P > 0.05$).

Table 2: Mean, lower, appear micro-shear bond strength values (MPa), standard deviation, and failure modes all groups

	Bond strength (MPa)			Failure modes, *n* (%)			
	Mean±SD	Upper	Lower	Adhesive	Cohesive (OC)	Cohesive (RC)	Mix
SBUs	49.3±10.4	71.3	32.1	9 (60)	2 (13)	1 (6)	3 (20)
SBU	47.4±10.3	72.2	36.1	10 (66)	2 (13)	1 (6)	2 (13)
AUs	34.3±9.5	52.1	22	10 (66)	1 (6)	2 (13)	2 (13)
AU	31.1±9.6	52.4	21.5	13 (87)	1 (6)	0	1 (6)
FUs	36.9±8.3	52.1	20.5	11 (73)	1 (6)	1 (6)	2 (13)
FU	21.5±5.3	32.1	12.8	13 (87)	0	0	2 (13)
CSEs	44.5±11.2	63.5	33.8	10 (66)	1 (6)	2 (13)	2 (13)
CSE	29.8±8.2	46.4	17.7	14 (93)	0	0	1 (6)
TSBs	34.6±9.1	52.4	23.5	14 (93)	0	0	1 (6)
TSB	30.5±9.4	45.3	18.6	13 (87)	0	1 (6)	1 (6)
SB2s	40.6±10.2	63.5	24.2	10 (66)	1 (6)	2 (13)	2 (13)
SB2	39.2±9.6	60.4	24.6	11 (73)	0	1 (6,7)	0

SD: Standard deviations, SBU: Single Bond Universal, SBUs; Silane + Single Bond Universal, AU: All-Bond Universal, AUs: Silane + All-Bond Universal, FU: Futurabond Universal, FUs: Silane + Futurabond Universal, CSE: Clearfil SE Bond, CSEs: Silane + Clearfil SE Bond, TSB: Tri-S Bond, TSBs: Silane + Tri-S Bond, SB2: Single Bond 2, SB2s: Silane + Single Bond 2, RC: Repair composite, OC: Original composite

SEM analysis of debonded surfaces showed that adhesive failure was the most frequently seen type of failure in all groups. Cohesive failure modes were rarely seen in all groups [Figures 1, 2 and Table 2]. The Chi-square test showed that there were no statistical differences among all the tested groups in terms of failure modes.

DISCUSSION

Recently, dental manufacturers have introduced multipurpose "universal one-bottle adhesives" for use as either self-etch or as etch and rinse adhesives. These adhesives are claimed by the manufacturers to be suitable for use for all kinds of hard tissue or materials. Since conventional adhesives required time-consuming additional surface procedures to repair fractured composite restorations, the repair protocol of composite restorations, using universal adhesives, may be useful when it comes to increasing the bond strength of composite resins.

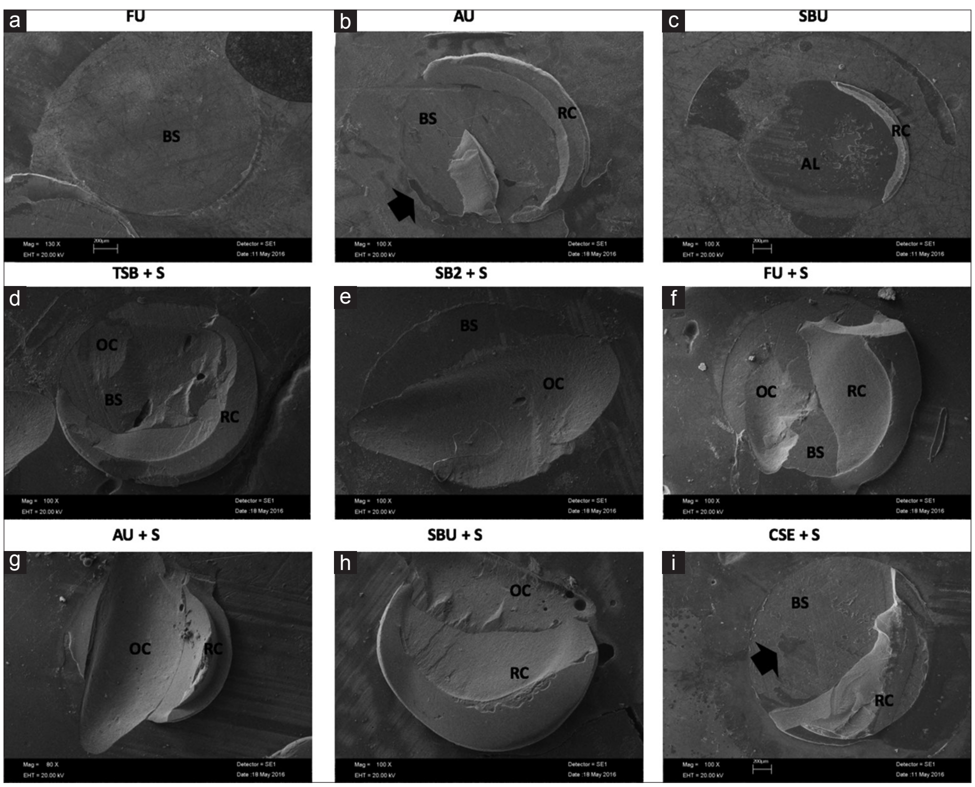

Figure 1: Scanning electron microscopy images of debonded surfaces (a) Adhesive failure, (b-d and i) Mix failure: small amount of adhesive (white arrows [b and i]) or repair composite remnants, (e and g) Cohesive failure in original composite: Fractured surface is mainly in original composite material; (f and h) Cohesive failure in repair composite: Fractured surface is mainly in repair composite material. FU: Futurabond Universal, AU: All-Bond Universal, SBU: Single Bond Universal, TSB: Tri-S Bond, SB2: Single Bond 2, CSE: Clearfil SE Bond, S: Silane, BS: Bonding surface, AL: Adhesive layer, RC: Repair composite, OC: Original composite

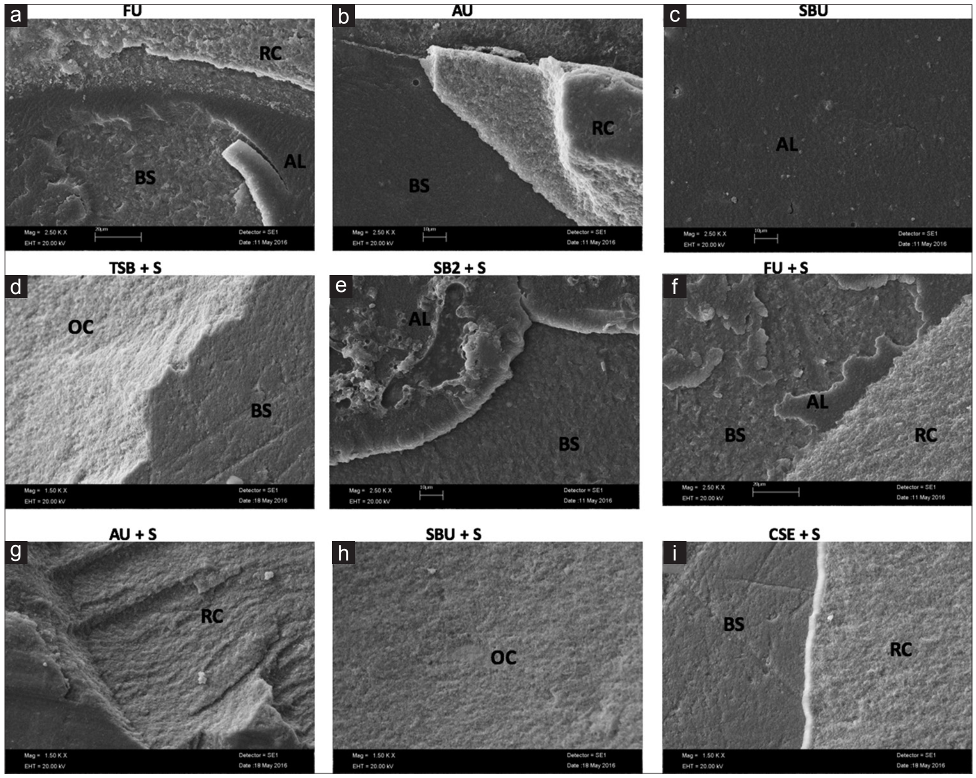

Figure 2: Scanning electron microscopy images of debonded surfaces (×2500). (a) FU: Futurabond Universal, (b) AU: All-Bond Universal, (c) SBU: Single Bond Universal, (d) TSB + S: Tri-S Bond + Silane, (e) SB2 + S: Single Bond 2 + Silane, (f) FU + S: Futurabond Univelsal + Silane (g) AU +S: All-Bond Universal + Silane (h) SBU + S: Single Bond Universal + Silane (i) CSE + S: Clearfil SE Bond + Silane, S: Silane, BS: Bonding surface, AL: Adhesive layer, RC: Repair composite, OC: Original composite

In the present study, universal adhesives did not improve the bond strength when used without silane. However, a universal adhesive containing silane, SBU increased the bond strength compared to conventional adhesives that were used without silane. When this adhesive was used without silane, it was also found to show higher bond strength than Clearfil TSB with silane and a similar bond strength to both CSE and SB2 that were used with silane. However, the other two silane-free universal adhesives, Futurabond U and ABU, did not increase the bond strength with or without silane. For this reason, the null hypothesis of the present study was rejected.

The manufacturers of universal adhesives who have added a silane coupling agent to the adhesives have considered the beneficial effects of the silane coupling agent in restoration repairs.[17,18] In a recent study, Staxrud and Dahl reported that the application of silane before adhesive procedures, or the use of a silane-containing bonding agent, Scotchbond Universal (known as "SBU" in some countries), increased the bond strength of aged composite resin.[19] Similarly, in the present study, SBU containing silane increased the bond strength. This can be explained by the fact that bifunctional silane molecules adhere to the surface after being hydrolyzed to silanol. Silanol groups form a polysiloxane network on the substrate, which finally reacts with the monomers of the resin composite, and consequently increases bond strength.[17]

Phosphate esters such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) found in universal adhesive systems, have many positive attributes, including the potential to bond chemically to metals, zirconia, and tooth tissues through the formation of nonsoluble Calcium salts. However, Futurabond U does not contain 10-MDP. This may be a reason why the bond strength values of Futurabond U were lower than those of SBU and ABU.

In the present study, although the addition of silane did not greatly affect the bond strength of two conventional adhesives, TSB and SB2, it increased the bond strength of CSE. Actually, it is expected that two adhesive systems (CSE, TSB) coming from the same manufacturer (Kuraray) with the addition of silane material may have better bond strength than the other adhesive materials used in the present study. However, the silane only increased the bond strength of CSE and did not change the bond strength of Clearfil TSB. This result may be due to the fact that CSE has an additional primer step containing a 10-MDP monomer that can bond chemically to metals. Furthermore, this additional step can increase the wettability of adherent surfaces. Furthermore, according to the manufacturers' instructions, the best procedure for composite repairs is to use a two-step self-etch adhesive system.

In the present study, a micro-shear test was used to evaluate the bond strength of the materials tested. This technique has some advantages because it is easy to perform, and does not need samples to be cut after bonding of the composite resin as in "micro-tensile" testing. This means that the bonding strength will not be reduced as a result of the slicing procedure.[20] More than one sample can be bonded to a test surface, therefore, it requires a fewer number of total samples for the study. The SEM examination of a larger number of samples at the same time may be easier than micro-tensile and macro-shear testing. The bonding area is smaller than that of the other shear bond test methods, and this may result in more "adhesive" type failure, and consequently more valid assessment. The "cohesive" fractures do not represent the clinically-relevant failure mechanism that occurs in real cavities. This particular problem can be prevented with micro-shear testing because the predominant failure during the investigation is also one of "adhesive" failure. Similarly, in the present study, the SEM images showed that the most common failure type related to the adhesive used.

The present study was conducted in an *in vitro* environment, and several factors such as oral fluids, occlusal forces, and thermal changes were not taken into account, and only composite samples were tested. Therefore, further *in vivo* and *in vitro* studies are needed to validate the results of the present study.

CONCLUSION

The effect of universal adhesives on composite repair depends on material selection. A silane-containing universal adhesive, SBU, can be safely used for composite repair, with and without silane. CSE containing silane can also be used in composite repair protocols. Under the tested conditions, the use of the other two universal adhesives is not suitable without silane, because they do not provide higher bond strength than conventional repair methods. Futurabond U cannot be used without silane.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

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