

Micro-shear bond strength of universal adhesives to caries-affected dentin; self-etch or etch-and-rinse strategy?

Universal adezivlerin çürükten etkilenmiş dentine mikro-makaslama bağlanma dayanımı; self-etch veya etch-and-rinse strateji?

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ABSTRACT

Introduction: The purpose of this study was to investigate the micro-shear bond strength of caries-affected dentin, using three universal adhesives either the self-etch or etch-and-rinse strategy.

Methods: A total of 180 resin composite cylinders (n=20 in each group) were bonded to caries-affected dentin tissue (A total of 90 carious human mandibular molar teeth were used, with 2 resin composite cylindrical samples on each tooth) using three universal adhesives [All Bond Universal (ABU), Futura Bond Universal (FU), (Single Bond Universal (SBU).] with, or without, acid etching and three conventional adhesives [Clearfil Tri-S Bond (TSB), Clearfil SE Bond (CSE), Single Bond 2 (SB2)]. After 5000 thermo-cycling rounds, a universal test device micro-shear bond strength test was realized at a crosshead speed of 0.5 mm/min to evaluate the bond strength of samples the debonding surfaces were assessed under the SEM. Two-way ANOVA and Tukey's Post-hoc tests were used to analyse the data.

Results: While acid etching increased the bond strength of FU significantly (p=0.003), it decreased the bond strength of SBU significantly (p=0.002) and did not change the bond strength of ABU (p=1.00).

Discussion and conclusion: The bond strength of universal adhesives on caries-affected dentin with, and without, acid etching depends on which universal adhesive system was used.

Keywords: Caries-affected dentin, micro-shear bond strength, resin composite, universal adhesive

ÖZ

Giriş ve Amaç: Bu çalışmanın amacı, üç universal adezivin self-etch ve etch&rinse olarak kullanılarak çürükten etkilenmiş dentine mikro-makaslama bağlanma dayanımını araştırmaktır.

Yöntem ve Gereçler: Toplam 180 adet kompozit silindir (her grupta n=20), self-etch ve etch&rinse olarak uygulanan üç universal adeziv, [All Bond Universal (ABU), Futura Bond Universal (FU), (Single Bond Universal (SBU).] ve üç geleneksel adeziv [Clearfil Tri-S Bond (TSB), Clearfil SE Bond (CSE), Single Bond 2 (SB2).] kullanılarak çürükten etkilenmiş dentine (Her bir dişte 2 adet rezin kompozit silindirik örnek olmak üzere toplam 90 insan alt çene azı dişi kullanıldı.) yapıştırıldı. Universal bir test cihazı kullanarak 5000 siklus termal döngü sonrasında örneklerin bağlanma dayanımını değerlendirmek için çapraz kafa hızı 0,5 mm/dk' da mikro-makaslama bağlanma dayanım testi uygulandı. Kopma yüzeyleri SEM altında incelendi. Veriler Two-way ANOVA ve Tukey's Post-hoc testleri kullanılarak analiz edildi.

Bulgular: Asitle pürüzlendirme, FU' nun bağlanma dayanımını önemli ölçüde artırırken (p=0.003), SBU' nun bağlanma dayanımını önemli ölçüde azalttı (p=0.002) ve ABU' nun bağlanma dayanımını değiştirmede (p=1.00).

Tartışma ve sonuç: Universal adezivlerin çürükten etkilenmiş dentin üzerindeki asitle pürüzlendirme ve asitle pürüzlendirme olmadan bağlanma dayanımı, hangi universal adeziv sistemin kullanıldığına bağlıdır.

Anahtar Kelimeler: Çürükten etkilenmiş dentin, mikro makaslama bağlanma dayanımı, rezin kompozit, universal adeziv

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INTRODUCTION

Most resin composites on the market need to be used with an adhesive system. In this respect, the quality and longevity of the restorative are achieved by providing an ideal micro-locking within the dental tissue.¹ Although the bonding mechanism of adhesive systems for sound dentin tissues is similar, the bonding behavior of adhesive systems may differ in CAD due to different histochemical structures.² Selective removal of caries tissue may be recommended as it will importantly lower the risk of pulp exposure and postoperative pulp indications, which is favorable in the treatment of deep caries.³ The intratubular deposition of calcium phosphate crystals in the periphery of the CAD, the high porosity rate of intertubular dentin, and the greater number of unsupported collagen fibrils may have a significant effect on bonding strength.⁴ In addition, due to the acidic environment in the superficial part of the CAD, the bonding is reduced by the activation of various matrix metalloproteinase enzymes. Briefly, bonding to the CAD tissue is more difficult than for those with healthy dentin.⁵ In recent years, manufacturers have presented to the dental market multi-purpose “universal adhesives”, that can be bonded to any kind of tooth tissue. They can be performed to the etch-and-rinse and self-etch strategy, and also there is no requirement for further surface pre-treatment. Unlike the majority of conventional adhesives, UA is much easier and faster to use and require less technical sensitivity.⁶

Recent research has examined several multimode adhesive systems and assessed how well etch-and-rinse and self-etch techniques function.⁷⁻¹⁰ A systematic review indicated that the etch-and-rinse approach constructs higher values for enamel; nevertheless, on dentin, for most of the UA systems contained, both approaches produced similar values.¹¹ Studies investigating the bonding of UA to CAD tissue are insufficient in the literature.^{2,4} In addition, studies were mostly carried out in artificial CAD tissue.^{2,4} However, the adhesion properties of new, multimodal adhesives to CAD have not yet been broadly presented. For this reason, the purpose of the current study was to investigate in which mode (etch-and-rinse and self-etch) the three UA's offer more successful bonding values on CAD and compare them with three conventional adhesives. The hypothesis of the study is that pre-etching will not make any difference in bond strength to CAD before the use of UA.

MATERIALS AND METHODS

The study protocol was approved by Nuh Naci Yazgan University Scientific Research and Publication Ethics Committee.

Tooth selection and preparation

In the present study, a total of 90 human mandibular molar teeth with occlusal or approximal carious were used. Attachments on the extracted teeth were cleaned with a scaler and ultrasonic bath and stored in a 5% Chloramine-T solution until used. The occlusal enamel surfaces of the teeth were cut under water-cooling with cylindrical diamond burs (Labcut 1010, Extec, Enfield, CT, USA) using a high-speed handpiece (#3146, KG Sorensen, SP, Brazil). For CAD, the samples were pulverized using a combination of the criteria of visual inspection and staining with 0.5 percent fuchsin dye until relatively firm, non-staining dentine was achieved.¹³ The exposed mid-coronal CAD surfaces were flattened for 60 seconds with 150 and 300 grit of silicon carbide paper, respectively, to obtain a standard smear surface. The teeth were then embedded in PVC rings up to 1 mm below of cemento-enamel junction, filled with auto-polymerized acrylic resin (İntegra, Birleşik Grup Dental, Ankara, Turkey) to expose the dentin surfaces and were randomly divided into nine groups in accordance with the adhesive procedure used.

Groups and adhesive procedures

The materials, composition, and application modes used in the current study are in Table 1.

- Group 1; Single Bond Universal (3M ESPE, Neuss, Germany) (SBU)
- Group 2; Acid + Single Bond Universal (3M ESPE) (SBUa)
- Group 3; Futurabond U (Voco, Cuxhaven, Germany) (FU)
- Group 4; Acid + Futurabond U (Voco) (FUa)
- Group 5; All Bond Universal (Bisco, Schaumburg, IL, USA) (AU)
- Group 6; Acid + All Bond Universal (Bisco) (AUa)
- Group 7; Single Bond 2 (3M ESPE, St. Paul, MN, USA) (SB2)
- Group 8; Clearfil Tri-S Bond (Kuraray, Okayama, Japan) (TSB)
- Group 9; Clearfil SE Bond (Kuraray, Osaka, Japan) (CSE)

The teeth in the acid-etched groups were treated with 37% orthophosphoric acid (Super Etch, SDI Inc, Bensenville IL, USA) for 15 seconds, then they were washed with an air-water spray for 15 seconds and air-dried. Each adhesive system was then applied to the dentin surfaces of all the teeth with the aid of a micro brush (Single Tim, Voco GmbH, Cuxhaven, Germany) in accordance with the manufacturer's instructions and polymerized in occluso-gingival direction for 20 seconds with an LED light tool (Valo, Ultradent Products Inc, South Jordan, USA) at 1000 mW/cm² output for the

micro-shear bond strength test, three composite resin samples (Charisma Heraeus Kulzer, Hanau, Germany) were attached to each dentin surface as bulk with the aid of a cylindrical plastic tube [Tygon, (0.75 mm inner

diameter and 1 mm height), Norton Performance Plastic Co., Cleveland, OH, USA] and then polymerized with the LED light tool for 20 seconds.

Table 1. Materials, composition, and application used in the present study

Materials	Composition	Application	
Charisma Heraeus Kulzer, Hanau, Germany Batch #010417A	Bis-GMA, TEGDMA, Barium aluminium fluoride glass, Silicium dioxide	1. Apply resin composite to surface, 2. 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	1. Apply primer to tooth surface and leave in place for 20 s 2. Dry with air stream to evaporate the volatile ingredients 3. Apply bond to the tooth surface and then create a uniform film using a gentle air stream 4. 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	1. Apply etchant for 15 s 2. Rinse for 10 s 3. Blot excess water 4. Apply 2–3 consecutive coats of adhesive for 15 s with gentle agitation 5. Gently air dry for 5 s 6. 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	1. Apply adhesive for 20 s 2. Air dry for more than 5 s 3. 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	Self-etch	Etch&Rinse
		1. Mix and stir thoroughly both liquids with the Single Tim applicator 2. Apply the adhesive homogenously to the surface and rub for 20 s using the Single Tim 3. Dry off the adhesive layer with dry, oil-free air for at least 5 s 4. Light cure the adhesive layer for 10 s	1. Etch the surfaces to be etched using a dental acid-etch agent based on phosphoric acid for 15 s 2. Aspirate the acid-etch agent, rinse with water for approximate 15 s 3. Dry off excess moisture with a gentle stream of air to produce a silky matte surface appearance Do not overdry the dentine 4. Apply adhesive as for the self-etch strategy
Single Bond Universal 3M ESPE, Neuss, Germany. Batch #535812	10-MDP phosphate monomer, Vitrebond, Copolymer, HEMA, BISGMA, dimethacrylate resins, Filler, silane, initiators, Ethanol, water	Self-etch	Etch&Rinse
		1. Apply the adhesive with the applicator to the entire surface and rub for 20 s 2. Dry gently for about 5 s until it no longer moves and the solvent has evaporated completely 3. Harden the adhesive with a curing light for 10 s	1. Apply etchant for 15 s 2. Rinse thoroughly with water and dry with water-free and oil- free air or with cotton pellets; do not overdry 3. Apply adhesive as for the self-etch strategy
All-Bond Universal Bisco, Schaumburg, USA Batch #1400007671	10-MDP phosphate monomer, HEMA, BISGMA, ethanol, water, initiators	Self-etch	Etch&Rinse
		1. Dispense 1–2 drops of ABU into a clean well 2. Apply two separate coats, scrubbing the preparation with a microbrush for 10–15 s per coat 3. Evaporate excess solvent by thoroughly air- drying for at least 10 s. Surface should have a uniform glossy appearance 4. Light cure for 10 s	1. Etch for 15 s 2. Rinse thoroughly 3. Remove excess water by lotting the surface with an absorbent pellet or high volume evacuation for 1–2 s, leaving the preparation visibly moist 4. Apply adhesive as for the self-etch strategy

Bis-GMA: Bis-phenol A diglycidylmethacrylate; HEMA: 2-hydroxyethyl methacrylate; TEGDMA: Triethyleneglycodimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; UDMA: Urethane Dimethacrylate; HEDMA: 1,6-hexanediol dimethacrylate; Al₂O₃: Aluminium oxide.

Thermal cycle and micro-shear bond strength test

All the teeth were exposed to thermal cycling 5000 times in distilled water from 5-55°C, 30 seconds of waiting time, and 5 seconds of transportation time. Then, the plastic tubes around the specimens were then removed with a scalpel. For micro-shear bond strength (μ SBS) testing, they were exposed to the universal testing machine (Instron Model 3345, Instron Corp., Canton, MA, USA). A 0.25-mm thick wire was formed into a loop shape and it surrounded the composite resin cylinder. The shear force was performed at a speed of 0.5 mm/min. The maximum bond strength is measured in Newtons (N), and its magnitude was determined by multiplying the bonding surface area (mm^2) by megapascals (MPa).

Scanning Electron Microscopy (SEM) analysis and determination of fracture types

Carbon paper from both sides was used to attach each tooth to the sample container. In order to add conductivity, a gold layer (90-135°A) was applied to the fractured sample surfaces using a sputter coater. They were then examined with an SEM device (LEO-440, Oxford, UK) at $\times 100$ and $\times 1500$ magnification, respectively. Fracture types were defined as “adhesive” (between dentin-adhesive resin, between composite resin-adhesive resin, or within adhesive resin); “cohesive in dentin” (a fracture in dentin); “cohesive in composite resin” (a fracture in composite resin); and “mixed” (adhesive and cohesive fracture, together).¹⁴

Statistical Analysis

The data were analyzed with the Kolmogorov-Smirnov test and the distribution was observed to be normal. The data analysis was using made by Two-Way ANOVA and Tukey HSD Post-Hoc tests. Fracture types also were analyzed via the Chi-square test.

RESULTS

The mean, maximum, and minimum micro-shear bond strength values, standard deviations, statistical differences, and failure modes of the groups are shown in Table 2. When the UA systems were evaluated within themselves, it was found that the bond strength of SBUa was statistically lower than SBU ($p=0.002$), but the bonding strength of FUa increased when compared to FU ($p=0.003$). Although a partial increase was observed in the bond strength of AUa compared to AU, this value was not statistically significant ($p=1.000$). Among all adhesive systems, CSE provided the highest bond strength value ($p<0.05$). Table 2 shows the fracture types, while Figures 1 and 2 show the SEM images of the fracture surfaces. In general, the most common type of failure observed in the groups was an adhesive fracture. The adhesive fracture was followed by mixed and cohesive fractures (CC and CD), respectively. According to the Chi-square test results, the groups did not show a statistically significant difference with regard to fracture types ($p>0.05$).

Table 2. Mean minimum, maximum bond strength values, standard deviations (SD) and failure modes of groups. Different superscript letters indicate statistical differences.

	SBU		FU		AU		CONVENTIONAL		
	SE ^{b,c}	E&R ^a	SE ^{a,b}	E&R ^c	SE ^c	E&R ^c	TRS ^{a,b}	CSE ^d	SB2 ^{b,c}
Bond Strength (Mpa)									
Mean	45,2	31,6	34,7	48,1	45,9	46,8	34,8	58,9	43,1
SD	9,1	7,3	8,4	9,6	8,3	10,4	8,1	11,2	8,8
Min	25,0	21,4	15,9	30,5	29,5	29,8	23,3	39,6	28,0
Max	60,7	48,5	46,6	61,1	59,7	58,5	48,1	78,7	59,5
Failure modes n (%)									
Adhesive	8 (53)	12 (80)	12 (80)	10 (67)	11 (73)	10 (67)	11 (73)	7 (47)	8 (53)
Cohesive in Dentin	1 (7)	0,0	0,0	0,0	1 (7)	1 (7)	1 (7)	1 (7)	1 (7)
Cohesive in resin composite	0,0	0,0	0,0	0,0	0,0	0,0	0,0	1 (7)	1 (7)
Mixed	6 (40)	3 (20)	3 (20)	5 (33)	3 (20)	4 (27)	3 (20)	6 (40)	5 (33)

SBU, Single Bond Universal; FU, Futurabond Universal; AU, All Bond Universal; TRS, Tri-S Bond; CSE, Clearfil SE Bond; SB2, Single Bond 2; SE, Self-etch; E&R, Etch&rinse.

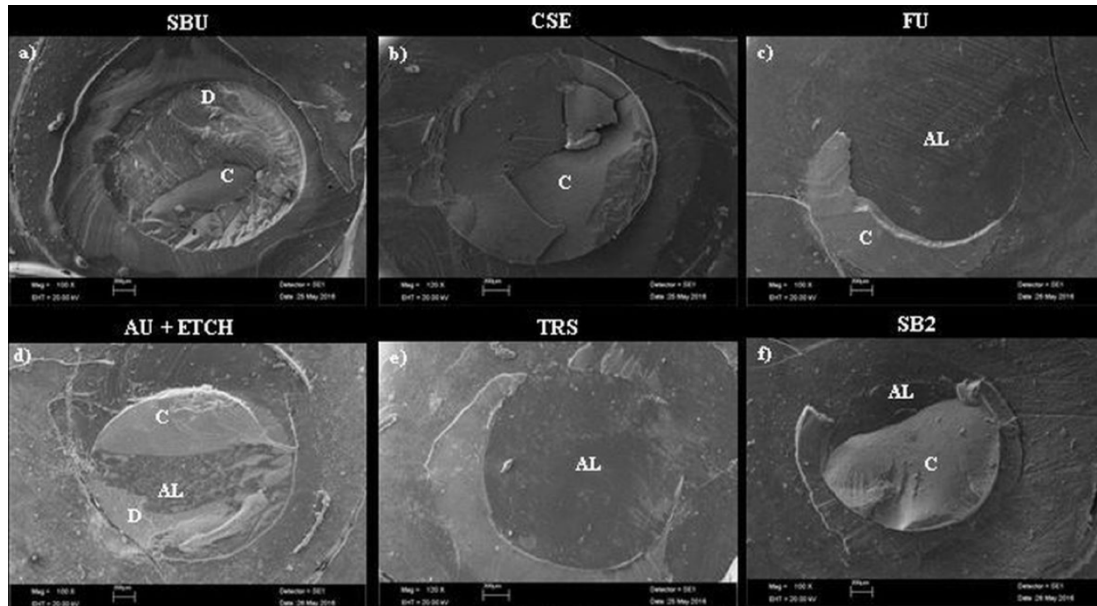


Figure 1. SEM images of debonded surfaces (x100 magnification): a) Cohesive failure in dentin, almost completely fractured dentin structure; b) Cohesive failure in composite, completely fractured composite structure; c) Mixed failure, small amount of composite remnant and adhesive layer; d) Mixed failure, fractured composite, dentin, and adhesive layer can be seen subsequently; e) Adhesive failure, failing between composite and adhesive resin; f) Cohesive failure in composite, largely the amount of composite structure and small amount of adhesive can be seen. FU, Futura Bond Universal; AU, All Bond Universal; SBU, Single Bond Universal; TRS, Tri-S Bond; SB2, Single Bond 2; CSE, Clearfil SE Bond; AL, Adhesive layer; C, composite; D, Dentin.

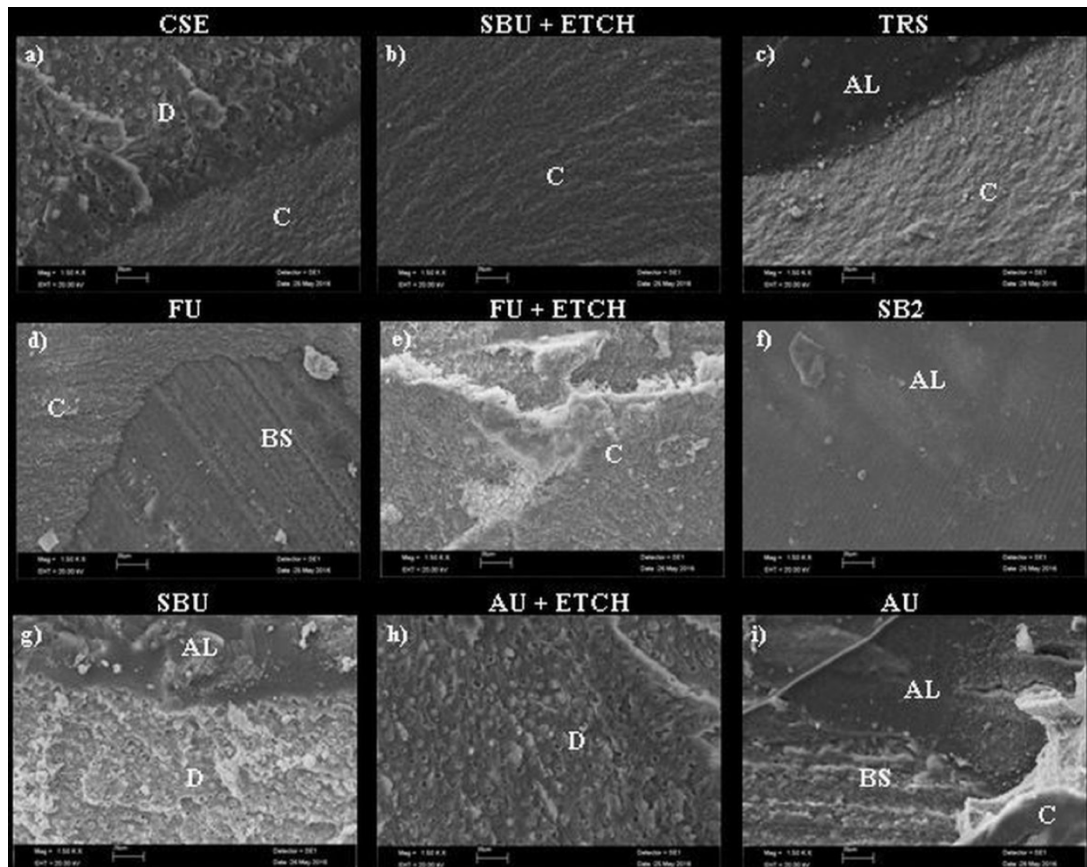


Figure 2. SEM images of debonded surfaces (x1500 magnification). FU, Futura Bond Universal; AU, All Bond Universal; SBU, Single Bond Universal; TRS, Tri-S Bond; SB2, Single Bond 2; CSE, Clearfil SE Bond; AL, Adhesive layer; C, composite; D, Dentin; BS, Bonding surface.

DISCUSSION

In the present study, the adhesive performance of the three UA, SBU, FU, and AU in the CAD with, or without, a pre-etching step was assessed. The micro-shear bond strength of these materials was tested by comparing these adhesives with three different traditional adhesive systems: Tri-S Bond, SB2, and SE Bond. According to the results of the study, when the SBU was used with acid, the bonding value was decreased, whereas FU increased the bond strength with pre-etching. The bond strength of AU did not change with acid etching. For this reason, the initial hypothesis of the study was rejected.

Investigators have reported that adhesive systems may exhibit different adhesion characteristics in CAD tissue compared to healthy dentin tissue, due to deposits of minerals that may affect monomer infiltration into dentin tubules.⁴ There are studies relating to the bonding performance of UA in healthy dentin tissues¹⁵⁻²⁰ and CAD tissue. However, when the literature was searched, it was seen that most of the studies conducted were on artificial caries-affected dentin. The current research is on caries-affected dentin in human teeth. Nicoloso et al.¹² investigated the bonding performance in CAD tissue. The researchers used Scotchbond Universal in their study and reported that this adhesive could be used safely in both the etch-and-rinse and self-etch approaches. On the contrary, the present study showed that the same adhesive (SBU) provided higher bonding values when used in the self-etch strategy. This discrepancy between the two studies may be due to the fact that Nicoloso et al.¹² used experimental artificial caries tissue in their study, whereas in the present study tissues with natural caries were used.

Nicoloso et al. found that the adhesive performance of SBU on CAD was similar to CSE and SB2.¹² This result is partially contradictory to the results of the current study because, in this study, CSE showed a remarkably higher bond strength among all the adhesives tested. This difference may have occurred due to the differences in the methods used between the present and their study. With respect to the results of the current study, FU increased the bond strength on CAD when used with acid-etch, unlike the SBU. The bond strength of AU did not change with acid-etch. These behavioral differences between the adhesives used in the study may be due to the independent chemical structures of each adhesive.

Dönmez et al.²¹ in their study reported that SBU adhesive had the highest bond strength. However, in this study, while there was no difference when applied as SBU adhesive self-etch, bond strength decreased when applied as etch-and-rinse. They also presented that ABU showed the lowest bond strength value. There was no difference in the present study. The reason for this is Dönmez et al.²¹ It may be that they applied Er: YAG laser to the CED surface.

When the literature was searched, no study was found on caries-affected dentin related to FU. In the current study, FU presented more successful bond strength values when applied as etch-and-rinse on CAD. Universal adhesives are composed similarly to traditional one-step self-etch adhesives. The majority of adhesives have specific carboxylate and/or phosphate monomers that chemically link to calcium in hydroxyapatite.²² Among these monomers, methacryloyloxydecyl dihydrogen phosphate (10-MDP) is included in most of the universal adhesives.²³ Although the FU does not contain MDP monomer, when applied as an etch-and-rinse on CAD, It offered bond strength values similar to those when SBU was applied as self-etch. Although universal adhesives have similar content, the proportions of the materials in their content differ. Therefore, the results of the present study can be explained in this way. On the other hand, Siqueira et al.²⁴ reported that when they applied FU as an etch-and-rinse in their study on healthy and eroded dentin, FU presented more successful results. The results of their study are similar to the results of the present study. Additionally, In the study of the same researchers, AU presented similar values to current study.

The micro-shear bond strength test was engaged in the current investigation to evaluate the adhesives' bonding performance. Compared to the micro-tensile and macro-shear approaches, this methodology is simpler to use. The test technique can be completed without risking degradation of the bond strength because there is no slicing step following the enforcement of the resin components. The bonding area is so small that more than one specimen can be placed on the dentin surface, which facilitates SEM inspection. The micro-shear bonding test also has the benefit of using fewer teeth than other procedures. Additionally, "adhesive" failure occurs more frequently than "cohesive" and "mixed" fracture types during the test because the bond surface area is less than in the macro-shear test. This makes the test more trustworthy.²⁵ In the current investigation, cohesive fractures were hardly detected, whereas adhesive fractures and mixed fractures were more constantly seen in groups with strong bonding. This finding demonstrates the reliability of the study's testing strategy.

CONCLUSION

Within the limitations of the present study, the following results may be concluded:

1. The bond strength of UA on CAD with, and without, acid etching depends on which UA system was used.
2. CSE showed higher bonding values than all the UA on the CAD.
3. Adhesive failure mode was most common for all adhesives followed by mixed failure.

Because the current study was performed under in vitro conditions, oral conditions such as occlusal forces,

thermal changes, and oral fluids were ignored. That's why additional in vitro and in vivo studies are needed.

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