

## Bond strength of five current adhesives to caries-affected dentin

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**Author(s):** Abdulkadir Sengün, Nymet Ünlü, Füsun Özer, Bora Öztürk  
University of Selçuk, Faculty of Dentistry, Konya, Türkiye

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### Introduction

All published reports on the performance of bonding systems have used normal dentin as the substrate, even though caries-affected and sclerotic dentin are often the clinically relevant bonding substrates. Little is known about the effects of different conditioner, etchant, and self-etching systems on the bond strengths of resin to caries-affected dentin. The structural or physical characteristics of the caries-affected collagen fibrils that are exposed by etching with different etchants may be different from that of normal dentinal collagen fibrils. The spaces between the collagen fibrils in normal dentin are occupied by normal calcium-deficient, carbonate-rich apatite (LeGeros 1991). In caries-affected inter-tubular dentin, the mineral occupying the interfibrillar spaces may be different from that of normal apatite due to cyclic demineralization-remineralizations. Indeed, the Knoop Hardness of caries-affected dentin indicate that it is softer, even though many of the tubules are occluded with mineral crystallites (Ogawa et al 1983, Nakajima et al 2000). It is theoretically possible for adhesive resin monomers to penetrate further into less mineralized, more porous demineralized dentin but not wet the collagen fibers very well. The complete penetration of resin monomers in to the demineralized dentin is essential to create strong adhesion as well as a perfect seal of the enveloped collagen fibers (Sano et al,1995). For normal dentin, many reports are available with regard to both the tensile properties and interfacial morphology between the resin and the hybrid layer. Little high-resolution SEM has been reported on the interfacial morphology of the junction between adhesive resin and caries-affected dentin and/or sclerotic dentin. Tagami (1999) showed morphological variations in the resin-impregnated layer that were dependent on whether the dentinal tubules were occluded or opened. The mineral phase of carious dentin is remodeled by repeated sequences of demineralization and remineralization, which usually produce occlusion of the tubules with mineral crystals.

### Objective

The purpose of this study was to test the hypothesis whether bonding to caries affected dentin will yield bond strengths that are lower than bonds made to normal dentin.

### Material and Methods

#### Specimen preparation

Sixty extracted human molars with approximal dentin caries extending approximately half way through the dentin were used in this study within one month of extraction. All teeth were stored at room temperature in physiological saline. The root of teeth were mounted in a 1,5x1,5 cm quadrangle mold using chemically cured acrylic resin. The teeth were mounted so that their long axis were perpendicular to the base of molds. The approximal surfaces with and without caries were prepared parallel to the long axis of the teeth to expose a flat dentin surface by using an Isomet saw under water (Buehler Ltd, Lake Bluff I L). For caries affected dentin, grinding was performed using the combined criteria of visual examination and staining with a caries detector solution (Kuraray Co, Ltd, Osaka, Japan).

#### Bond strength analysis

The teeth were randomly divided in to five groups, each containing 12 teeth. Five commercially available adhesive systems and their resin composite materials were used (Table1).

The flat dentin surfaces of teeth were hand-polished with 600 grit silicon carbide abrasive paper under running water. The surfaces were examined after polishing to ensure that its orientation was not altered. After the completion of the bonding procedures, according to the manufacturers' instructions (Table 1, Figure 1) composite resins were added to the surface by packing the material into cylindrical shaped plastic matrixes with an internal diameter of 2.5 mm and height of 3 mm. Excess composite was carefully removed from periphery of matrixes with an explorer. Bonding agents and composites were cured with an HILUX Curing Light for required seconds. The intensity of light was 400 mW/cm<sup>2</sup> at least. Specimens were then stored in distilled water at 37°C for 10 days before bond strength testing.



Figure 1: The materials used in the study

Bonding Systems	Components	Tipi	Composite	Procedures
Prime Bond 2.1 DENTSPLY (PB)	PENTA, TEGDMA, Bis GMA, Fluoride, Acetone	One Bottle	Prisma TPH	a - 20s. c - 5s. g - 10s. e, c
One Coat Bond COLTENE/ Whaledent inc. USA (OCB)	Collene Etchant 15: Water, phosphoric acid 15% gel former HEMA, HPMA, Glycerindimethacrylat, Polyalkenoat methacrylat, UDMA, Amorphoas Silica	One Bottle	Synergy	a - 30s. b - 20s. c - 2s. f - 20s. c - 2s g - 30s
Clearfil SE KURARAY (CSE)	Primara MDP, HEMA, Hydrophilic dimethacrylate, di-camphorquinone, N,N-Diethanol-p-toluidine and water Adhesive: MDP, Bis-GMA, HEMA, Hydrophobic dimethacrylate, di-camphorquinone, N,N-Diethanol-p-toluidine and Silanated colloidal silica	Self etch sistem	Clearfil AP-X	a - 20s. c f g - 10s.
Etch&Prime 3.0 DEGUSSA AG Geschäftsbereich Dental Hanau (EP)	Catalyst: Pyro-phosphate, HEMA, Initiators, stabilizers Universal: 2-HEMA, ethanol, distilled water, stabilizer	Self etch sistem	Degufill mineral	d e - 30s. c - 5s g - 30s
Solid Bond KULZER (SB)	Esticid-20FG Solid Bond P: Aceton, Methacrylate, Maleic acid Solid Bond S: Bis GMA	Total etch sistem	Pokallite	a - 15-30s. b - 15s. c e - 30s. c, f, c g - 40s

Table 1. Manufacturers, components and application procedures of the dentin bonding systems used in the study

For shear bond testing, the specimens were mounted in a universal testing machine (Testometric Micro 500, England). A Knife-shaped apparatus attached to a compression load cell and traveling at a cross head speed of 1 mm/min was applied to each specimen at interface between tooth and composite until failure occurred. The maximum load (N) was divided by the cross-sectional area of the bonded composite posts to determine shear bond strength in Mpa.

### Fracture analysis

Fracture analysis were performed using an optical stereomicroscope (Olympus SZ4045 TRPT, Japan). Failures were classified as: cohesive if more than 80 % of resin was found remaining on the tooth surface, adhesive if less than 20 % of the resin remained on the tooth surface, or mixed if certain areas exhibited cohesive fracture while other areas exhibited adhesive fracture.

### Statistical Analysis

The data were analyzed by Kruskal-Wallis one way analysis of variance: Mann-Whitney U multiple comparison test was used to determine specific differences between groups. Wilcoxon Rank Tests were used to test shear bond strength differences in between caries-affected and normal dentin in the same tooth.

### Results

The results of the shear bond testing were summarized in Table 2 and Figure 2. For different types of bonding systems, ANOVA analysis showed significant differences in mean values of bond strengths for both caries-affected ( $p=0.020$ ) and normal dentin ( $p=0.000$ ). Among the materials applied to caries-affected dentin, CSE, SB, and EP showed higher bond strength than OCB and PB ( $p < 0.05$ ). SB, EP, and OCB showed statistically similar values ( $p < 0.05$ ). Among the materials applied to normal dentin, CSE had the highest bond strength ( $p < 0.05$ ). Also, SB and EP showed higher bond strength than OCB and PB ( $p < 0.05$ ).

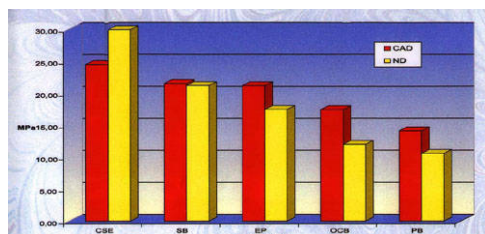


Figure 2. Mean shear bond strength values of materials (MPa) to caries-affected (CAD) and normal dentin (ND)

CSE, SB, and EP had bond strengths which were not much different for caries-affected and normal dentin ( $p > 0.05$ ). However, bond strength of OCB and PB to caries-affected dentin were significantly higher than to normal dentin ( $p < 0.05$ ).

Materials	n	Significance		
		Caries-Affected dentin	Normal Dentin	
		Means(SD)(MPa)	Means(SD)(MPa)	
CSE	12	24.49±5.38	NS	29.91±8.95
SB	12	21.49±9.15	NS	21.17±5.41
EP	12	21.19±9.17	NS	17.45±6.21
OCB	12	17.43±9.78	0.006	11.99±10.15
PB	12	14.10±7.94	0.034	10.57±4.56

Table 2. Mean shear bond strength values of materials (MPa) to caries-affected and normal dentin

Group	n	Cohesive	Adhesive	Mixed
Clearfil SE Bond	24	11(7)	8	5
Caries-Affected dentin	12	6 (5)	3	3
Normal Dentin	12	5 (2)	5	2
Solid Bond	24	(6)	16	2
Caries-Affected dentin	12	(4)	7	1
Normal Dentin	12	(2)	9	1
One Coat Bond	24	7 (4)	17	-
Caries-Affected dentin	12	5 (4)	7	-
Normal Dentin	12	2	10	-
Prim& Bond 2.1	24	-	23	1
Caries-Affected dentin	12	-	11	1
Normal Dentin	12	-	12	-
Etch&Prime	24	4 (1)	17	3
Caries-Affected dentin	12	3 (1)	6	3
Normal Dentin	12	1	11	-

Table 3. Modes of failure

In general, a greater percentage of the fractures were adhesive at the tooth-composite junction. The fracture patterns of the specimens of the materials involved are given in Table 3. The macroscopic mode of failure for bonding systems SB, OCB, PB and EP appeared to be adhesive in nature. That is the bond failed in the dentin/composite interface. With system CSE, most of the failures appeared to be cohesive failure of the composite and dentin, although about 33% of the bond failures showed some adhesive failure and about 21% of the bond failures showed some mixed failure (Figure 3a, b, c).



Figure 3a. Fracture surface from shear bond test with SB Bond showing cohesive failure in CAD

Figure 3b. Fracture surface from shear bond test with SE Bond showing mixed failure in ND

Figure 3c. Fracture surface from shear bond test with PB Bond showing adhesive failure in ND

## Discussion and Conclusions

In this report, the shear bond strengths of CSE, SB, EP, OCB, and PB (2.495.38, 21.499.15, 21.199.17, 17.439.78, and 14.107.94 MPa respectively) to caries affected dentin and (29.918.95, 21.175.41, 17.456.21, 11.9910.15, and 10.574.56 MPa respectively) to normal dentin indicate that the later two system were more sensitive to caries affected dentin than the former three system. The results of this study indicated that there was no significant difference between the bond strength obtained on normal and caries affected dentin.

Sano et al (1995) tested the hypothesis that resin infiltration of demineralized dentin can restore its tensile properties to those of mineralized dentin. The results of their study indicate that some adhesive resins, after infiltrating demineralized dentin, can restore and even exceed the ultimate tensile strength of mineralized dentin.

Nakajima et al (1995) tested whether bonding to caries-affected dentin is inferior to bonding to normal dentin, and that the quality of the hybrid layer plays a major role in creating good adhesion. Bonding to normal dentin with either All Bond 2 (26.9±8.8 MPa) or Clearfil Liner Bond II (29.5±10.9 MPa) showed tensile bond strengths higher than those to caries-affected dentin (13.0±3.6 MPa and 14.0±4.3 MPa, respectively). The tensile bond strengths obtained with Scotchbond Multi-Purpose were similar in normal and caries-affected dentin (20.3±5.5 MPa and 18.5±4.0 MPa, respectively).

In another recent study, it was found that Scotchbond Multi-purpose Plus the bond strengths to dry, normal dentin were only half as strong (21±10 MPa) as those made to moist, normal dentin (42±9 MPa). There was no significant difference between bonds made to normal vs. caries affected dentin by means of the moist technique (42±9 vs. 48±4 MPa, respectively) (Nakajima et al 1999).

There is concern that some acidic conditioners may not be strong enough to adequately etch sclerotic or caries-affected dentin.

Nakajima et al (2000) found that with One-Step following etching with 10% phosphoric acid showed lower ( $p<0.05$ ) tensile bond strengths to caries-affected dentin compared to normal dentin (36.9±8.0 MPa vs 47.7±6.5 MPa respectively). This difference disappeared when using 32% phosphoric acid (49.7±6.1 MPa vs 45.0±7.2 MPa respectively). Bonds made to caries-affected dentin with Single bond were always lower than bonds to normal dentin regardless of the strength of the phosphoric acid.

In this study, even though two one-bottle bonding systems performed better bonding values on the caries effected dentin than they performed on the normal dentin. Both total-etch and self-etch bonding systems, which had similar values on caries affected and normal dentin, showed higher bond strength values than one-bottle systems. These results indicate that both total etch and self etch bonding systems were more successful on both normal and caries affected dentin than one-bottle systems.

## Conclusion

The type of dentin and its histologic structure play an important role during bonding performance of adhesive resin materials. And also chemical composition of a bonding system can alter bonding mechanism.

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## Abbreviations

Bis GMA : bisphenyl-glycidyl-methacrylate

HEMA: 2-Hydroxyethyl methacrylate

MDP: 10-Methacryloyloxydecyl dihydrogen phosphate

HPMA: Hydroxypropylmethacrylate

PENTA: Pentaacryloyldipentaerythrytol phosphoric acid

TEG-DMA: triethyleneglycol dimethacrylate

*This Poster was submitted on 03.02.01 by Dr. Abdülkadir Sengün.*

## Correspondence address:

*Dr. Abdülkadir Sengün*

Selcuk Üniversitesi Dis Hekimligi

Fakültesi, Dis Hastalikari ve Tedavisi A.D

Tedavi Bölmünü 42079, Kampüs/Konya

Türkiye

# BOND STRENGTH OF FIVE CURRENT ADHESIVES TO CARIES-AFFECTED DENTIN

ABDULKADİR ŞENGÜN, NİMET ÜNLÜ, FÜSUN ÖZER\*, BORA ÖZTÜRK

(Selçuk University, Faculty of Dentistry, Konya, TÜRKİYE)

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## INTRODUCTION

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The purpose of this study was to test the hypothesis whether bonding to caries affected dentin will yield bond strengths that are lower than bonds made to normal dentin.

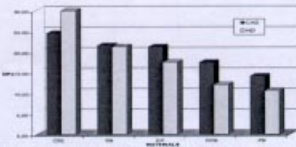


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CSE, SB, and EP had bond strengths which were not much different for caries-affected and normal dentin (p>0.05). However, bond strength of OCB and PB to caries-affected dentin were significantly higher than to normal dentin (p<0.05).

Table 2. Mean shear bond strength values of materials (MPa) to caries-affected and normal dentin

Material	n	Caries-Affected dentin Mean (SD) (MPa)	Significance	Normal Dentin Mean (SD) (MPa)
CSE	12	12.4 (4.8)	NS	16.0 (2.8)
SB	12	12.1 (4.8)	NS	16.0 (17.5)
EP	12	12.1 (4.8)	NS	16.0 (17.5)
OCB	12	17.4 (9.7)	0.006	20.0 (10.8)
PB	12	14.3 (7.4)	0.024	18.0 (11.5)

Groups connected by a bar are not significantly different. NS: No significance between caries affected and normal dentin

Table 3. Modes of failure

Group	n	Cohesive	Adhesive	Mixed
Shear Bond	60	3	3	54
Caries-Affected dentin	12	0	0	12
Normal Dentin	12	0	0	12
Self Bond	24	0	0	24
Caries-Affected dentin	12	0	0	12
Normal Dentin	12	0	0	12
One Coat Bond	24	7	0	17
Caries-Affected dentin	12	4	0	8
Normal Dentin	12	3	0	9
Bond 2	12	0	0	12
Caries-Affected dentin	12	0	0	12
Normal Dentin	12	0	0	12
ES&AP Bond	24	4	0	20
Caries-Affected dentin	12	3	0	9
Normal Dentin	12	1	0	11

\*Numbers in parentheses denote specimens with visible cracks in the dentin surface. In general, a greater percentage of the fractures were adhesive at the tooth-composite junction. The fracture patterns of the specimens of the materials involved are given in Table 3. The macroscopic mode of failure for bonding systems SB, OCB, PB and EP appeared to be adhesive in nature. That is the bond failed in the dentin/composite interface. With system CSE, most of the failures appeared to be cohesive failure of the composite and dentin, although about 32% of the bond failures showed some adhesive failure and about 21% of the bond failures showed some mixed failure (Figure 3a, b, c).



Figure 3a: Fracture surface from shear bond test with SB Bond showing cohesive failure in CAD. Figure 3b: Fracture surface from shear bond test with SE Bond showing cohesive failure in ND. Figure 3c: Fracture surface from shear bond test with PB Bond showing adhesive failure in SB.

## DISCUSSION

In this report, the shear bond strengths of CSE, SB, EP, OCB, and PB (2,435.36, 21,409.15, 21,169.17, 17,439.78, and 14,107.84 MPa respectively) to caries affected dentin and (29,918.25, 21,175.41, 17,456.21, 11,970.15, and 10,574.56 MPa respectively) to normal dentin indicate that the latter two system were more sensitive to caries affected dentin than the former three system. The results of this study indicated that there was no significant difference between the bond strength obtained on normal and caries affected dentin.

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Nakajima et al (1995) tested whether bonding to caries-affected dentin is inferior to bonding to normal dentin, and that the quality of the hybrid layer plays a major role in creating good adhesion. Bonding to normal dentin with either All Bond 2 (28,963.8 MPa) or Clearfil Liner Bond II (28,561.0 MPa) showed tensile bond strengths higher than those to caries-affected dentin (13,262.5 MPa and 14,034.3 MPa, respectively). The tensile bond strengths obtained with Scotchbond Multi-Purpose were similar in normal and caries-affected dentin (20,345.5 MPa and 18,564.0 MPa, respectively).

In another recent study, it was found that Scotchbond Multi-Purpose Plus had the bond strengths in dry, normal dentin were only half as strong (21,110 MPa) as those made to moist, normal dentin (42,525 MPa). There was no significant difference between bonds made to normal vs. caries affected dentin by means of the moist technique (42,525 vs. 48,246 MPa, respectively) (Nakajima et al 1995).

There is concern that some acidic conditioners may not be strong enough to adequately etch sclerotic or caries-affected dentin. Nakajima et al (2000) found that with One-Step following etching with 10% phosphoric acid showed lower (p<0.05) tensile bond strengths to caries-affected dentin compared to normal dentin (29,918.25 MPa vs 47,745.5 MPa respectively). This difference disappeared when using 32% phosphoric acid (49,745.5 MPa vs 45,037.2 MPa respectively). Bonds made to caries-affected dentin with Single bond were always lower than bonds to normal dentin regardless of the strength of the phosphoric acid.

In this study, even though two one-bottle bonding systems performed better bonding values on the caries affected dentin than they performed on the normal dentin, both total-etch and self-etch bonding systems, which had similar values on caries affected and normal dentin, showed higher bond strength values than one-bottle systems. These results indicate that both total etch and self-etch bonding systems were more successful on both normal and caries affected dentin than one-bottle systems.

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## MATERIALS AND METHODS

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Figure 1. The materials used in the study

Table 1. Manufacturers, components and application procedure of the dentin bonding systems used in the study

Bonding Systems	Components	Etch	Composite	Procedure
One Coat Bond (OCB)	One Coat Bond 2, HEMA, Anionic	One Coat Bond 2	Prime 200	1-20, 2-24, 3-24, 4-24
Self Bond (SB)	One Coat Bond 2, HEMA, Anionic	One Coat Bond 2	Prime 200	1-20, 2-24, 3-24, 4-24
Clearfil Liner Bond II (CLB)	Clearfil Liner Bond II, HEMA, Anionic	Clearfil Liner Bond II	Prime 200	1-20, 2-24, 3-24, 4-24
ES&AP Bond (ES&AP)	ES&AP Bond, HEMA, Anionic	ES&AP Bond	Prime 200	1-20, 2-24, 3-24, 4-24
Single Bond (SB)	Single Bond, HEMA, Anionic	Single Bond	Prime 200	1-20, 2-24, 3-24, 4-24

Procedure: 1- acid etching 15 sec at 37°C; 2- rinsing; 3- application of adhesive; 4- light cure

### Abbreviations

ES&AP: Etch-and-seal adhesive system  
 HEMA: 2-hydroxyethyl methacrylate  
 SBP: 10-hydroxybutyl methacrylate  
 HEMA: hydroxyethyl methacrylate  
 PHTCA: Phosphoric acid  
 TEO-DMA: triethylene glycol dimethacrylate

For shear bond testing, the specimens were mounted in a universal testing machine (Testometric MTR 200, England). A Kofler-shaped apparatus attached to a compression load cell and traveling at a cross head speed of 1 mm/min was applied to each specimen at interface between both and composite until failure occurred. The maximum load (N) was divided by the cross-sectional area of the bonded composite posts to determine shear bond strength in MPa.

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Fracture analysis were performed using an optical stereomicroscope (Olympus SZ4545 TRPT, Japan). Failures were classified as: cohesive if more than 80 % of resin was found remaining on the tooth surface, adhesive if less than 20 % of the resin remained on the tooth surface, or mixed if certain areas exhibited cohesive fracture while other areas exhibited adhesive fracture.

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The data were analyzed by Kruskal-Wallis one way analysis of variance. Mann-Whitney U multiple comparison test was used to determine specific differences between groups. Wilcoxon Rank Tests were used to test shear bond strength differences in between caries affected and normal dentin in the same tooth.

## RESULTS

The results of the shear bond testing were summarized in Table 2 and Figure 2. For different types of bonding systems, ANOVA analysis showed significant differences in mean values of bond strengths for both caries affected (p<0.02) and normal dentin (p<0.005). Among the materials applied to caries-affected dentin, CSE, SB, and EP showed higher bond strength than OCB and PB (p<0.05). SB, EP, and OCB showed statistically similar values (p>0.05). Among the materials applied to normal dentin, CSE had the highest bond strength (p<0.05). Also, SB and EP showed higher bond strength than OCB and PB (p<0.05).

