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Shear bond strength of different adhesive systems to normal and caries-affected dentin

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Original Article

Abstract

BACKGROUND AND AIM: According to the effect of the adhesive and substrate type on the bond strength, examination of the adhesive is required in all aspects. The aim of this study was to evaluate the shear bond strength of different adhesive systems to normal dentin (ND) and caries affected dentin (CAD) in permanent teeth.

METHODS: Thirty extracted molars with small occlusal caries were selected**.** After preparation and determination of ND and CAD by caries detector, teeth were divided into three groups and treated with one of the two tested adhesives: Single Bond 2 (SB2), Scotchbond Universal with etch (SBU-ER), and Scotchbond Universal without etch (SBU-SE). Then composite (Filtek Z-250 XT) were attached to the surfaces and cured. After water storage (24 hours) and thermocycling (500 cycles 5-55 °C), bond strength was calculated and failure modes were determined by stereomicroscope. The data were analyzed by one-way ANOVA and post-hoc test [Tukey HSD (honest significant difference)] and with $P < 0.050$ as the level of significance.

RESULTS: Only SBU-ER had significantly higher shear bond strength than SBU-SE in ND ($P = 0.027$) and CAD ($P = 0.046$). Bond strength in SBU-ER the highest and in SBU-SE had the lowest amounts in CAD and ND. There was no significant difference in each group between ND and CAD.

CONCLUSION: The 2-step etch-and-rinse adhesive (SBU-ER) had higher bond strength to ND and CAD than the self-etch adhesive (SBU-SE).

KEYWORDS: Dentin Bonding Agent Normal, Caries-Affected Dentin, Shear Bond Strength Test

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Dental caries is the most common pathological change in dentin. Cardiology research indicated that the caries dentin has two layers: outer layer [caries infected dentin (CID)] and inner layer [caries affected dentin (CAD)]. The outer layer contains bacteria and is highly demineralized. In fact, some irreversible changes occurred in the collagen fibrils, but the inner layer, CAD, has the potential of remineralization. It is partially demineralized, without bacteria and with reversible collagen changes.[1,2] So, it should be maintained during the clinical treatments.[3] Although a wide area of the cavity floor after caries removal is CAD,[1,3] whereas the most in vitro researches conducted on the normal dentin (ND) and because of the histological difference between them, study on CAD is necessary. CAD contains the collagen fibrils with lower collagen cross-linkages, bigger apatite crystallites,[4,5] and wider crystal spaces in the intertubular dentin. The matrix of the peritubular dentin in CAD contain

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mucopolysaccharide or glycoprotein molecules which can interfere with the adhesion.[4]

The interface between the adhesive and CAD in comparison with ND, is wider and more complicated,[4] and some mineral deposits exist in the dentin tubules of CAD such as White lock, it avoid complete penetration of resin monomers in tubules and formation of the resin tags due to the acid resistance of obturated tubules.[5] Therefore, the formed hybrid layer on CAD is not only thick but also poorer in quality.[4] In addition, large quantities of water in deeper demineralized areas in CAD interfere with the penetration of resin monomers and bonding process. Also, CAD contains the materials which interfere with the bonding process by producing and developing the free radicals thus leading to the lesser polymerization of the resin monomers.[1]

The created hybrid layer by adhesive systems in CAD becomes thick but porous.[4] Also, CAD's hardness is lower than ND because of more porosity in the intertubular dentin,[6] which is due to the loss of mineral materials. This demineralization and porosity lead to the cohesive weakness. The bond strength of CAD is lower than that of ND due to the microstructural changes in dentin.[4]

Another effective factor in the bond strength between the adhesive resin and dentin is the type of adhesive system. The current adhesive systems are classified to etch-and-rinse (ER) and self-etch (SE) systems.[7] ER systems are technique sensitive because of the multiple steps.[8] In these systems, phosphoric acid 35-37% causes a complete removal of the smear layer, collagen fibrils exposure, and dentin demineralization. It is shown that there is a discrepancy between the depth of demineralization and the resin penetration.[1] In SE adhesives, simultaneously demineralization and resin penetration is occurred, and they are less technique sensitive.[8] Shear bond strength test was one of the most widely used mechanical properties test and is a maximum force at adhesive joint before fracture occurs.[9]

There are several studies of shear bond strength in CAD and ND with controversial results,[7,10] which some studies were shown higher bond strength of ND[11,12] or CAD[10,13] in ER adhesives, but in some other studies, there did not see significant difference in bond strength of them.[8,14]

Scotchbond Universal (SBU) is a novel adhesive which the manufacturer claims that it has less technical sensitivity due to the polyalkenoic acid compound[15] and applicable in both ER and SE methods. Thus, there were not done any study about its efficacy on CAD, the purpose of this study was the evaluation of shear bond strength of this novel adhesive in ER and SE methods and one ER adhesive [Single Bond 2 (SB2)] in CAD and ND. The null hypothesis is there was no significant difference in shear bond strength of the mentioned adhesives is ND and CAD.

Methods

This experimental study was done on the 30 freshly human extracted molar teeth which had occlusal caries that extending approximately halfway through the dentin. After removing calculus and soft tissue, occlusal enamel and the caries of occlusal surface (by using diamond fissure burs, Tiz Kavan, Iran) were removed under running water to reach superficial flat dentinal surface. The samples were excluded if the obtained area reaches to deeper dentin. Affected caries removal was done by visual examination and a comparatively sharp explore and staining by using caries detector (Kuraray Medical Inc., Sakazu, Kurashiki, Okayama, Japan).

According to the manufacturer's recommendations, caries detector solution was applied on the surface for 10 seconds and then washed. Therefore, the soft and dark-red stained and the harder pink-stained dentin were considered as CID and CAD, respectively. After above mentioned procedure, there was enough amount of ND and CAD area in each sample. Efficiency of CAD area was examined by a periodontal probe (at least 2 mm in diameter). To create flat levels and standard smear layer, a 600 grit silicon carbide sand paper was used. Then, the teeth were mounted up to the cementoenamel junction in the self-cure acrylic resin (Acropars, Iran) in a way that the occlusal surface of the teeth was located horizontally.

Finally, the samples were randomly divided into three groups for each type of adhesive. Group 1: Adper™ SB2 (2-step ER, 3M, ESPE, USA). Group 2: Scotchbond Universal without etch (SBU-SE) (1-step SE, 3M, ESPE, USA). Group 3: Scotchbond Universal with etch (SBU-ER) (2-step ER, 3M, ESPE, USA). The adhesives were used according to the manufacturer instruction (Table 1).

Curing was done by a quartz-tungsten-halogen unit (Demetron LC, Kerr, USA) with 600 mw/cm^2 intensity (The device intensity was checked by radiometer). Then, the micro hybrid composite (Filtek Z-250 XT, Shade: B1, 3M, ESPE, USA) was placed on the bonded area by a clear plastic cylindrical tube (2 mm diameter and 2 mm height) in two layers and each layer was cured for 20 seconds. After removing the tubes, samples were stored in distilled water in an incubator at 37 °C for 24 hours then were thermocycled for 500 cycles (5-55 °C).

The shear bond strength test (Blade type) was done by the Universal Testing Machine (Testometric M350-10 CT, Lancashire, United Kingdom) with 0.5 mm/min crosshead speed with a chisel-shaped device. The shear bond strength was calculated in megapascal.

Mode of failure was identified by two examiners by observed the debonded surface levels separately by a stereomicroscope (Olympus, DP12, Germany) at × 40 magnification. Finally, the type of failure (cohesive/adhesive or mixed) was identified. To compare the shear bond strength in each group used one-way ANOVA analysis and post-hoc test [Tukey HSD (honest significant difference)]. $P < 0.050$ was set as the level of significance.

Table 1. Composition and application techniques of the tested materials

Material	Type	Manufacturer	Composition	Application technique
Adper™ Single Bond 2	2-step ER adhesive	3M, ESPE, USA	Etchant: 35% phosphoric acid Adhesive: Bis-GMA, HEMA, ethanol, water, ethanol	Etching: 15 seconds, Rinse:10 seconds, blot dry, apply 2 coats of adhesive with gentle agitation, gently air thin: 5 seconds, cure for 20 seconds
Scotchbond Universal	1-step SE or 2-step ER adhesive	3M, ESPE, USA	10-MDP, HEMA, Vitrebond copolymer, filler, ethanol, water, initiators, silane	SE mode: adhesive 20 seconds, gently air thin: 5 seconds, cure: 20 seconds ER mode: Scotchbond etchant Gel: 15 seconds, Rinse: 10 seconds, blot dry, adhesive 20 seconds, gently air thin: 5 seconds, cure: 20 seconds
Filtek Z-250 XT	Light curing nano Hybrid resin composite	3M, ESPE, USA	BIS-GMA, UDMA, BIS-EMA, PEGDMA, TEGDMA, zirconium, silica	-
Ultra Etch	Etching agent	Ultradent, USA	35% phosphoric acid	-

ER: Etch-and-rinse; SE: Self etch; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: Hydroxyethyl methacrylate; UDMA: Urethane dimethacrylate; TEGDMA: Triethylene glycol dimethacrylate; Bis-GMA: Bisphenol A-glycidyl methacrylate; PEGDMA: Polyethylene glycol dimethacrylate; Bis-EMA: Ethoxylated bisphenol-A glycol dimethacrylate

Table 2. Shear bond strength data in MPa and mean percentage of failure mode

Adhesive	Normal dentin MPa (mean ± SD)	A (%)	M (%)	CC (%)	CD (%)	P	Caries-affected dentin MPa (mean ± SD)	A (%)	M (%)	CC (%)	CD (%)
SB2	20.16 ± 9.61*	70	30	0	0	0.32 (NS)	20.17 ± 9.53*	20	30	20	30
SBU-SE	18.93 ± 3.23**	80	0	0	2	0.91 (NS)	18.84 ± 7.83**	30	60	10	0
SBU-ER	26.88 ± 4.63***	10	60	10	20	0.44 (NS)	27.70 ± 5.71***	0	90	10	0

A: Adhesive; M: Mix; CC: Cohesive in composite; CD: Cohesive in dentin; SB2: Single Bond 2; SBU-SE: Scotchbond Universal without etch; SBU-ER: Scotchbond Universal with etch; MPa: Megapascal; NS: Not significant; SD: Standard deviation
*, ** and *** for each raw, means designated by the same sign are not significantly different (P > 0.050), NS: Not significantly different between normal and caries-affected dentin (P > 0.050)

Results

Results of the present study showed significant differences in ND (P = 0.022) and CAD (P = 0.038) groups for types of adhesive systems. There were no significant differences in each group between ND and CAD. SBU-ER had significantly higher bond strength than SBU-SE in ND (P = 0.027) and CAD (P = 0.046), but there was no significant statistical difference between groups SB2 and SBU-SE, also between groups SB2 and SBU-ER in ND and CAD (P > 0.050). Most of the failure mode in groups SB2, SBU SE, and SBU-ER, were adhesive, adhesive, and mix, respectively in ND. Also in CAD, the major of failure mode in all groups were mixed. The results of bond strength and failure modes are shown in table 2.

Discussion

Tooth loss is directly related to oral health, and the protection of tooth tissue is one of the main goals of restorative dental treatments. On the other hand, because of a large area of the bonding substrate after removal of carious dentin, is CAD that can remineralize, keep this tissue seems necessary in dental treatment.[1] Considering the effect of the substrate on the performance bond,[4] the performance of different adhesive systems to ND and CAD was evaluated in this study.

The results of the present study show that Only SBU-ER had significantly higher shear bond strength than SBU-SE in ND (P = 0.027) and CAD (P = 0.046). Bond strength in SBU-ER had the highest and in SBU-SE had the lowest amounts in CAD and ND. There was no significant difference in each group between ND and CAD. The null hypothesis was partially confirmed in some aspects.

In this study, to differentiate between CAD or CID, caries detector (Kuraray Medical Inc., Sakazu, Kurashiki, Okayama, Japan) was used beside the visual and tactile examination. Yokota et al. in a study showed that after a complete rinse of the surface after caries detector usage, no undesirable effect on the bond strength of adhesives to the ND was observed.[16]

In our study, there was no significant statistical difference in bond strength between ND and CAD in each group. Based on the study of Wei et al. the bond strength of SB2 in ND and CAD was the same.[4] There are some studies on the ER adhesives with the similar results too.[14,17] On the other hand, Arrais et al.[11] reported that the bond strength to ND in SB was higher than CAD. Unlike Nakornchai et al.[13] and Tosun et al.[10] study which indicated higher bond strength of CAD than ND. Difference in result can be related to the difference in the technique sensitivity of ER adhesives,[7,17] as well as the difference in the methodology.[18] Also in the study of Omar et al., it is shown that thermocycling led to the significant decreasing of the bond strength in ND, but not in CAD in the ER adhesives.[19] Due to the minerals that occluded the tubules in CAD, further penetrating of water to the interface was avoided.[20] This can explains the similarity between the two group results in our study.

In ER adhesives, phosphoric acid can dissolve the mineral deposits in tubules and intertubular dentin of CAD which leads to a better penetration of the resin monomers.[21] Scanning electron microscopy images present the resin tags with the lateral branches and tubular anastomosis that probably increases the bond strength. The hybrid layer in CAD is thicker than that of ND. The resin tags are shorter and more disorder.[7] The partial demineralization of the intertubular dentin in CAD which allows deeper penetration of the phosphoric acid or the acidic monomers rather than ND can be a factor of the thicker hybrid layer in CAD.[17]

According to the results of this study, the bond strength of SBU SE was not different significantly in CAD and ND groups, which is similar to many studies.[6,8,13] Methacryloyloxydecyl dihydrogen phosphate (MDP) monomer presence in SBU compound leads to durable chemical bond with tooth structure. In CAD, the formation of water tree and water droplets occurs less than ND due to the closing tubules by the minerals.[21] However, there are some studies indicating a considerable decrease in the bond strength in CAD rather than ND by applying self-etch adhesives.[11,12,19] In addition to lack of resin tag formation in CAD, a decrease in the modulus of elasticity and the cohesive strength of dentin can lead to this decrease.[22]

The difference between SBU and SB in the relative replacement of the dimethacrylate monomers with 10-MDP monomer, which provides the acidity and demineralization capability of SBU simultaneously.[23] In addition, the presence of Vitrebond™ Copolymer of polyalkenoic acid in SBU make a desirable bond in dentin in the states of either dry or wet conditions,[24] which is applicable as SE and ER according to the manufacture's claim. The hybrid layer thickness in SBU-ER type was measured in 2.0-2.6 µm by wet bonding and 1.4-2.4 µm by dry bonding, whereas it is 0.2-0.4 µm by self-etch method.[23]

In our study, SBU-ER in ND and CAD, showed a signification higher bond strength than SBU-SE, which is in agreement with some other studies,[3,17] and related to unique composition of SBU.[24] Using the phosphoric acid causes a significant improvement of the interface morphology by forming a thicker hybrid layer and longer resin tags. Removing the smear layer and smear plug by using acid leads to the easy penetration of the adhesives.[5]Also, Munoz et al. study showed a higher bond strength in the ER method than the self-etch method in SBU.[25] The reason of the lower bond strength in all in one adhesives is mentioned by Perdigao et al. as the lack of sufficient polymerization of the adhesive due to oxygen inhibition by the so thin adhesive layers.[20]

Using all-in-one adhesives in multilayers can increase the bond strength which is resulted from various mechanisms simultaneously. The first layer of adhesive, which the beginner etching of dentin substrate may become buffer quickly by hydroxyapatite and the extra layers of adhesives may increase the etching ability by increasing concentration of the acid content. In addition, using more layers make the adhesive layer thicker (so avoid of oxygen inhibition in the whole thickness) with higher mechanical properties and resistance to polymerization shrinkage stresses.[5]

Mode of failure in the shear tests is influenced by the mechanic of test and the stress distribution during force applying and does not show the bond efficiency necessarily. In this study, the difference in mode of failure may result from the different mechanical properties of adhesives, as well as the different attributes of the formed interfaces in adhesives.[4] In ND, the most common failure mode in SBU-SE was adhesive (80%) and in CAD the most common failure mode in SBU-ER were mixed (90%) that can explain the results in their groups. The limitation of this study was difficulties in samples collection with at least 2 mm in diameter CAD and same depth of dentin in all samples.

Conclusion

Within the limitations of this study, it was concluded that shear bond strength in CAD was similar to ND in self-etch and etch-and-rinse adhesives.

Conflict of Interests

Authors have no conflict of interest.

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