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Influence o f E tching M ethods o n t he Shear B ond Strength o f a Universal Adhesive System to Sound and Artificial Caries-Affected Dentin

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ABSTRACT

This study tested the bond strength of a universal adhesive system to both caries-affected (CAD) and sound dentin after using different etching techniques.

Methods: Forty premolars were wet grinded to expose flat occlusal midcoronal dentin. Specimens were equally divided according to the substrate into sound and (CAD) specimens which were exposed to pH- cycling for 14 days. Single Bond™ universal adhesive was used to bond each dentin substrate either after no etching or etching of dentin, after which, resin composite was used for build-up. These specimens were then tested for shear bond strength (SBS). The results were then analyzed by two-ways ANOVA.

Results: Single Bond™ universal bonded to sound dentin had statistically significantly higher SBS mean values compared to CAD. Single Bond™ applied after separate etching step of dentin showed a statistically higher SBS compared to self-etching method when bonded to sound dentin.

Conclusion: Separate etching improved bonding to sound dentin and did not influence the bond strength of tested universal adhesive to CAD.

1. INTRODUCTION

Dentin is a challenging substrate for bonding as it is a dynamic structure, has non- uniform permeability, and has inherent moisture. In current literature, studies are abundant concerning bonding to normal dentin. Recently, more efforts have been made to have a conservative approach, through selective caries removal, maintaining tooth substrate, and preventing avoidable pulp exposure. This method entails leaving behind caries-affected dentin (CAD), after removing caries-infected dentin. Ultimately, the composite resin is bonded to the cavity floor, which is mainly formed of CAD. It can be expected that CAD would affect the performance of dental adhesives, due to its structural and morphological variations. [1, 2]

The performance of the newly developed universal adhesives applied to dentin with various etching procedures, depending on the clinical situation, is still a source of debate. [3] Therefore, the current study was conducted to assess how bonding to CAD using a Single Bond Universal adhesive system applied in different adhesion protocols will affect the shear bond strength in comparison with sound dentin.

The null hypotheses of this study were that: (1) There was no difference in the shear bond strength at a resin-dentin interface in caries affected dentin and sound dentin. (2) There was no effect of dentin etching methods on the resin-dentin shear bond strength.

2. MATERIALS AND METHODS

Materials tested

One universal adhesive system [Single Bond™ universal adhesive (3M ESPE, Germany)] and one resin composite [Filtek™ Z250^{XT} nano-hybrid universal resin composite (3M ESPE, USA)] were utilized in the current study.

Teeth Selection

Forty freshly extracted sound human upper premolars were selected from age group patients ranging from 18 to 25 years. Teeth were cleaned from periodontal fibers and soft tissues under running tap water using sharp hand scaler. Teeth were also examined for cracks, caries, fracture or any pathological abnormalities using magnifying lens (25X) and only teeth free from any defect were selected. Teeth were then kept at 4°C soaked in distilled water, for a maximum period of three months after extraction with a weekly change of the distilled water. [4]

Grouping of Specimens

The 40 selected premolars were divided into two main equal groups (n=20) according to the dentin substrate condition; either sound dentin or

artificially caries-affected dentin. Each main group was divided into two equal subgroups (n=10), according to the etching method utilized: either etch-and-rinse or self-etch.

Specimens preparation:

Roots of all teeth were cut 2mm below cemento-enamel junction by a double-sided diamond disc mounted to low-speed handpiece. The contents of the pulp chamber were then removed with a broach. Occlusal enamel was removed by wet grinding using a 240-grit silicon carbide (SiC) abrasive paper mounted in a bench grinder machine to expose midcoronal dentin. Checking the remaining dentin thickness was done using precise caliper. Only specimens of remaining dentin thickness 2 mm were used in the current study. Each exposed dentin surface was finished using wet SiC 600-grit paper for 60 seconds in a circular motion to create a standardized smear layer.[5]

Each dentin specimen was embedded in the acrylic resin up to the cemento-enamel junction, with the prepared occlusal surface facing outward. All specimens were immediately immersed in distilled water after complete acrylic resin polymerization until being used.

Development of caries-affected dentin (artificial caries):

Half of the dentin specimens (n=20) were exposed to cariogenic challenge by pH cycling to produce artificial caries lesion. PH cycling procedure and demineralizing and remineralizing solutions preparation were done according to **Nicoloso et al**. [6]

Application of the tested universal adhesive system with different etching methods on both sound and caries-affected dentin substrates:

For standardization of the bonded area, all dentin specimens were covered with an adhesive tape that provided a hole of 2 mm diameter, located at the center of dentin surface. The universal adhesive was applied either in self-etch or etch-and-rinse adhesion protocols according to the assigned group.

Self-etch: Single Bond™ universal adhesive was applied to the assigned dentin area for bonding using a micro brush and rubbed for 20 seconds. The adhesive layer was air thinned using gentle oil free compressed air for 5 seconds to evaporate the solvent. The adhesive layer was light cured for 10 seconds according to the manufacturer's instruction using LED light curing unit at intensity 1000 mW/Cm^2 .

Etch-and-rinse: Specimens assigned for this group were first acid-etched with 37% phosphoric acid gel (Meta Biomed, Germany) for 15 seconds followed by rinsing thoroughly with water for 15 seconds. Specimens were then blot dried. Single Bond™ universal adhesive was then applied to the assigned dentin area for bonding as mentioned in the self-etch adhesion protocol.

Application of the nano-hybrid resin composite

After the bonding procedure has been completed, the resin composite was packed in a cylindrical transparent plastic tube of inner dimensions (2 mm diameter and 3 mm height) which act as a mold during resin composite application. [7] Resin composite was packed in 2 increments 1.5 mm each, then each increment was light-cured for 10 seconds according to the manufacturer instructions using LED light curing unit of 1000 mW/cm^2. After removing the plastic tubes all resin composite cylinders were checked using magnifying glass lens (25X) to detect any defects. If any imperfection was found in any resin composite cylinder, the specimen was discarded. Each restored specimen was stored in 15 ml artificial saliva (pH=7) in an incubator at 37 °C for 48 hours until testing. Artificial saliva composition was prepared according to **Pashley et al**. [8]

Shear bond strength (SBS) test:

Shear bond strength test was carried out for all bonded specimens using a universal testing machine (Lloyd Instruments Ltd;,model LRX-plus;, Fareham, UK) . A chisel-shaped shearing blade with a 0.5 mm wide sharp edge was aligned parallel with the flat dentin surface of the bonded specimen. The load cell control system was then adjusted to apply load force of (5N) by chisel on dentin-restoration interface at a crosshead speed of 0.5 mm/min until failure occurs. The control system and its associated software recorded the maximum force needed to de-bond each specimen in Newton (N), which was automatically calculated to express the shear bond strength records on output device in Mega pascal (MPa).[9]

Statistical analysis

The shear bond strength mean and standard deviation values were calculated for each group. Data were explored for normality using Kolmogorov-Smirnov and Shapiro-Wilk tests, data showed parametric (normal) distribution. Independent sample t-test was used to compare between two groups in non-related specimens. Two-way ANOVA tests were used to test the interactions between different variables. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

3. RESULTS

Results of Two-way analysis of variance ANOVA for the effect of dentin substrate, and etching methods as independent variables and their interaction on the shear bond strength (MPa) showed that both dentin substrate and the etching method had a statistically significant effect at *P-value*< 0.001 and *P-value*= 0.019 respectively. The interaction between the two variables had no statistically significant effect at P-value= 0.929. Mean and standard deviation (SD) of the shear bond strength values for different groups were shown in Table 1.

Table 1:

The mean, standard deviation (SD) values of Shear bond strength (MPa) of different groups

Variables	Shear bond strength				p-value
	Etch-and-rinse		Self-etch		
	Mean	SD	Mean	SD	
Sound dentin	13.56	3.09	9.33	1.97	**0.008***
Caries affected dentin	8.66	2.52	7.52	2.66	**0.348ns**
p-value	**0.001***		**0.036***		

*; significant (p<0.05)
ns; non-significant (p>0.05)

4. DISCUSSION

Results of the current study showed that bonding the universal adhesive to caries-affected dentin had statistically significant lower SBS (MPa) values than sound dentin. This was in accordance to previous studies. [6, 7, 10, 11] This might occur because during the demineralization process caries-affected dentin lost an amount of its mineral components from its intertubular dentin. This part lost minerals was replaced by water, which negatively affect the bond quality. Moreover, caries-affected dentin undergoes ultra-morphological variations, such as decrease in organic content with lowering in collagen cross-linkages, intertubular dentin increased porosities, and degradation of

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collagen fibrils. Furthermore, the peritubular dentin matrix in caries-affected dentin contains noncollagenous protein, which may interfere with adherence to the substrate. [6, 11, 12]

Another reason for the low resin-dentin bond strength to caries-affected dentin, was that application of phosphoric acid or acidic monomers to it could lead to a deeper demineralization due to the substrate lower mineral content compared to normal dentin, resulting in a more thicker and porous hybrid layer compared to the hybrid layer formed on sound dentin, that negativity affect the bond strength. [7]

Single Bond™ universal adhesive applied in the etch-and-rinse mode on sound dentin showed a statistically higher resin-dentin bond strength compared to the self-etch method. Some studies found that this etching step advances the resin infiltration into sound dentin, forming longer resin tags and thicker hybrid layers. [13- 15] On the other hand, other studies [6, 16] found no change in Single Bond™ universal bond strength when applied to sound dentin, regardless of which strategy was used. This discrepancy in the results might be because different testing conditions were utilized in these studies.

The superiority of Single Bond™ universal on the etched sound dentin substrate might be related to the fact that the affinity of HEMA in Single Bond™ universal seems to be increased when combined with ethanol solvent. This formula allowed a decrease in the viscosity of the adhesive, improving its wetting on the etched dentin surface and maintain the expanded form of the collagen fibrils after the evaporation of solvents. Thus, monomer's penetration was enhanced into the dentin substrate. Resulting in a strong micromechanical interlocking and effective formation of a resin-demineralized dentin hybrid layer. [14, 17]

In addition; the Single Bond™ universal contained Vitrebond copolymer. This polyalkenoic acid copolymer could create Ca-polyalkenoate complexes at the outer region of the hybrid layer and within the dentinal tubules superficial micrometers which might stabilize the bonded interface by presenting water stability and a stress-relaxing outcome. [14, 17]

Finally, the first null hypothesis in the current study which stated that there was no difference between resin-dentin shear bond strength of caries affected dentin and that of sound dentin, was rejected. While regarding the second hypothesis it was partially rejected, as there were differences between the etching methods when bonded to sound dentin only.

5. CONCLUSION

1. CAD was negatively influenced the bond strength of Single Bond™ universal in both etching protocols.
2. Etch-and-rinse adhesive protocol improved the bond strength of Single Bond™ universal to sound dentin.

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