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Bond Strengths of a 1-Step Self-etching System to Caries-affected and Normal Dentin

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Clinical Relevance

The 1-step self-etching adhesive had significantly higher mean bond strength to normal dentin than to caries-affected dentin. Although Single Bond had a similar tendency, bonds to normal dentin and caries-affected dentin were not significantly different.

SUMMARY

This study was designed to evaluate the bond strengths of a 1-step self-etching system and a 2-step "etch and rinse" adhesive system to caries-affected dentin and normal dentin. In addition, the micromorphology of the adhesive interfaces was analyzed using scanning electron microscopy (SEM). Extracted human molars with occlusal caries that had been stored frozen were ground in order to expose the caries-affected dentin and surrounding normal dentin. The teeth were then bonded using either Adper Prompt L-Pop or Single Bond (3M ESPE) and restored with Filtek Z250 (3M ESPE). After storage in water for 24 hours at 37°C, the teeth were sectioned, prepared for microtensile bond strength test and tested in tension at a crosshead speed of 1-mm/minute. After debonding of the interfaces, microhardness of the dentin underlying the interface of all specimens was measured. The thickness of the hybrid layers was observed under SEM. The results of this study indicate that the bond strength of Adper Prompt L-Pop adhesive was significantly higher to normal dentin than to caries-affected dentin (*p*<0.05) and that the bond strength of Single Bond to both normal and caries-affected dentin was not significantly different (*p*>0.05). Additionally, the thickness of the hybrid layers produced by both adhesive systems was thicker for caries-affected dentin.

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INTRODUCTION

The development of recent adhesive restorative materials has brought about great changes in the philosophies of cavity preparation. Extensive cavity preparations have been replaced by more conservative techniques, with removal of the infected, permanently damaged caries tissue only, preserving the bacteria-free, caries-affected dentin or sclerotic dentin.[1] Caries-affected dentin contains intrinsic properties that are different from normal dentin, such as reduced permeability because of formation of whitlockites within the dentinal tubules and a partially demineralized intertubular dentin.[2-3] Ideally, in order to maximally preserve the tooth substrate, the clinical bonding substrate should be caries-affected dentin. However, during restorative preparations, it may be very difficult to avoid cutting into normal dentin due to the irregular shape of the caries lesion.

Clinically, although most adhesion studies are performed on normal dentin, most substrates are not normal dentin. Clinicians must deal with caries-affected dentin, cervical sclerotic dentin or even intrinsic differences between coronal and radicular dentin. Conventional testing methodologies are not applicable to such clinically relevant substrates due to the limited size and irregular shape of the abnormal dentin. The microtensile bond test[4-5] has been used to test the bond strengths of adhesive resin systems to caries-affected dentin and cervical sclerotic dentin.[6-10]

It has been previously reported that, although bond strengths to normal dentin were similar, bond strengths to caries-affected dentin of a 3-step "etch and rinse" system were significantly higher than to those of a 2-step self-etching system.[10] This difference was attributed to differences in acidity of the phosphoric acid versus mild primer and, consequently, the degree of demineralization of the caries-affected or sclerotic dentin. These differences would allow different degrees of adhesive penetration into demineralized dentin.[9-11]

This study evaluated bond strengths of a 1-step self-etching system with a ph of 1.2 (data from manufacturer) and a 2-step "etch-and-rinse" adhesive system to caries-affected and normal dentin. In addition, the micromorphology of the adhesive interfaces was analyzed using scanning electron microscopy (SEM). The null hypothesis was that there would not be any significant difference in bond strengths of the 1-step self-etching system to caries-affected and normal dentin.

METHODS AND MATERIALS

Extracted human molars with occlusal caries that had been stored frozen were used in this study. The teeth were thawed, cleaned of debris and the occlusal surfaces were ground flat to remove enamel and expose middle caries and normal dentin. Five teeth were used for each group. The occlusal soft, stainable carious dentin was ground off with 600-grit silicon carbide paper under running water. The underlying relatively hard caries-affected non-staining dentin was exposed and flattened to the same level as the adjacent normal dentin. The soft, stainable carious dentin was detected using Caries Detector (Kuraray Co, Ltd, Japan), which is a solution of 1% acid red 52 in a propylene glycol base.

The adhesives used in the study were Adper Prompt L Pop self-etching adhesive system (3M ESPE Dental Products, St Paul, MN, USA) and Single Bond (3M ESPE) as the control. The dentin surface treatment, bonding and light-curing procedures were carried out as recommended by the manufacturer (Table 1). A "crown" was built incrementally with Filtek Z250 (3M ESPE) resin composite to a height of 3-5 mm over the bonded surface using approximately 2-mm increments that were light-cured for 40 seconds each. All specimens were stored for 24 hours in tap water at 37°C.

The teeth were then sectioned into 0.7 mm-thick slices and, before trimming with a fine diamond, the caries-affected dentin or normal dentin, was carefully determined by both visual and light microscopic observations. The total number of slabs (n) were dependent on the diameter of the caries-affected dentin on each tooth (approximately n=5-8 slabs per specimen). The interfaces were then trimmed into an hourglass shape using a fine diamond bur (to produce a 1 mm^2 area) for microtensile bond testing. All specimens were subjected to tensile forces at a crosshead speed of 1-mm/minute using an EZ-Test testing machine (Shimazu Co, Kyoto, Japan).[4] Microtensile bond strengths (MPa) were deter-

Table 1: *Manufacturer's Instructions for the Use of Single Bond and Adper Prompt L Pop*

Adhesive System	Single Bond	Adper Prompt L Pop
Instructions	1) Apply Scotchbond Etchant to enamel and dentin for 15 seconds.	1) Apply adhesive with a rubbing motion for 15 seconds.
	2) Rinse for 10 seconds.	2) Gently but thoroughly air-dry to remove the aqueous solvent.
	3) Blot excess water using a cotton pellet or mini-sponge. Do not air dry.	3) Apply a second coat (no waiting time for the second layer).
	4) Immediately after blotting, apply 2-3 consecutive coats of adhesive for 15 seconds with gentle agitation using a fully saturated applicator.	4) Gently but thoroughly air-dry to remove the aqueous solvent.
	5) Gently air-thin for 5 seconds to evaporate solvent.	5) Light cure for 10 seconds.
	6) Light-cure for 10 seconds.	

mined by dividing the fracture load by the cross-sectional area of the interface. The thickness of the slabs and the width of the trimmed surfaces were measured using a digital caliper (Mitutoyo, Kawasaki, Japan).

After the tensile testing procedure, the debonded specimens were fixed in 10% neutral buffered formalin. They were embedded in epoxy resin and polished to high gloss using ascending grits of diamond pastes. Between each grit of diamond paste, the specimens were ultrasonically cleansed in a water bath. Knoop hardness numbers (KHN) of the subsurface were measured at 25-50 µm below the interface at 50 grams load for a dwell time of 15 seconds (Micromet 2100, Buehler, Lake Bluff, IL, USA). A mean Knoop hardness number for each specimen was calculated as an average of 3 Knoop hardness numbers obtained from each specimen.

After the dentin hardness measurement, the specimens were again lightly polished, subjected to acid/base treatment and sputter-coated with gold. With the Knoop hardness numbers in hand, the morphology of the caries-affected and normal resin-dentin interface was observed and selected. Excluded were the specimens exhibiting a combination of normal and caries-affected dentin beneath the bonded area.

All data were statistically analyzed by 1- and 2-way Analysis of Variance (ANOVA) and by Fisher's PLSD test.

RESULTS

Table 2 summarizes the mean bond strengths and Knoop hardness numbers of all the groups. Although the bond strengths of Single Bond to normal dentin showed a tendency to be higher than to caries-affected dentin, the difference was not statistically significant (p=0.188). In contrast, the bond strengths of Adper Prompt L Pop self-etching adhesive to normal dentin were significantly higher than to caries-affected dentin (p=0.006). For each substrate (normal or caries-affected), the bond strengths of Single Bond and Adper Prompt L Pop self-etching adhesive were similar (p>0.05).

The Fisher's PLSD analysis results for the 2-way ANOVA are summarized in Table 3. Two-way ANOVA revealed that bond strengths to normal dentin were significantly higher than to caries-affected dentin (p=0.0046). In addition, there was no statistically significant difference between adhesives (p=0.1515).

The Knoop hardness of normal dentin ranged between 54 and 74 KHN, and with caries-affected dentin, it ranged between 26 and 45 KHN. Specimens with carious dentin (KHN<25) or a mixture of hardness values (normal and caries-affected dentin) were deleted in order to ensure accuracy of the study.

The thickness of the hybrid layers of the caries-affected dentin specimens for both adhesive systems was greater that that of normal dentin. In general, the thickness of the hybrid layers for the self-etching adhesive specimens was significantly smaller than that of the Single Bond specimens (0.5–0.9 ± 0.3 µm and 3.0–4.2 ± 0.6 µm, respectively).

The resin tags produced by Single Bond showed hybridization of the peritubular dentin close to the tubule opening and were wide and funnel-shaped. On the other hand, the resin tags produced by the Adper Prompt L Pop adhesive system, though funnel-shaped, were thin and had less aggressive hybridization of the peritubular dentin.

Table 2: *Mean Bond Strengths (standard deviation) and Mean Knoop Hardness Numbers*

	MPa (SD; n)	KHN
PLP Normal Dentin	52.0 (17.5; 15)[a]	66.0
PLP Caries-affected Dentin	37.3 (9.7; 12)[b]	38.9
Single Bond Normal Dentin	43.3 (14.1; 12)[a,b]	68.2
Single Bond Caries-affected Dentin	36.1 (8.2; 12)[b]	41.2

Values with the same superscript letter are statistically similar (p>=0.05).

Table 3: *2-way ANOVA Table for MPa with the Factors Adhesive and Dentin*

	Effect: Adhesive (SB, PLP)	Effect: Dentin (Normal, Ca-affected)
Mean Difference	-5.533	11.299
Critical Difference	7.637	7.637
***p*-value**	0.1515	0.0046

DISCUSSION

Caries progression occurs as a function of a dynamic cyclic process of demineralization and remineralization. By definition, carious dentin is comprised of 2 layers: (a) the outer infected layer, which is a very soft tissue infected with bacteria and is totally degraded and (b) an inner affected layer, which is a harder tissue affected and sclerosed by the caries process.[12] This caries-affected dentin is uninfected and remineralizable and is usually included in portions of the preparation, which, in turn, also contains portions of normal dentin. Nevertheless, it is of great concern that previous studies have reported that the bond strength to caries-affected dentin was lower than that of normal dentin.[7,10]

Although caries-affected dentin is such a clinically relevant substrate, conventional testing methodologies would only allow for the evaluation of relatively flat, large surface areas. Therefore, bond strength studies were routinely performed on normal human or bovine dentin. However, the microtensile bond test[4] revolutionized bond strength studies, since it could be modified for testing in various dental substrates.

The bond strengths of Adper Prompt L Pop were significantly higher to normal dentin than to caries-affected dentin, and Single Bond showed a similar trend despite a non-statistical significance, probably due to the large standard deviation. These results are similar to those of Nakajima and others,[10] who reported that the bond strengths of All-Bond 2 and Clearfil Liner Bond II were higher to normal dentin than to caries-affected dentin and that Scotchbond Multi-Purpose showed similar bonds to normal and caries-affected dentin. On the other hand, the results of this study differ from those reported by Yoshiyama and others[7] and Nakajima and others,[13] who reported that the bond strength of Single Bond to normal dentin was significantly higher than to caries-affected dentin. The results of this study showed a tendency towards higher results for normal dentin; however, the difference was not statistically significant, possibly due to the large standard deviation, operator variability and/or technique sensitivity of Single Bond.

The Knoop hardness numbers of dentin underlying the tested interface was measured to ensure accuracy of the visual inspection. Microhardness of normal dentin ranged from 54-74 KHN and that of caries-affected dentin ranged from 26-45 KHN, both of which are in accordance with previously reported studies.[1,6,10,14] Therefore, specimens that contained caries in affected dentin (<25 KHN) or both normal and caries-affected dentin within the tested area were deleted from the study. Caries-affected dentin is known to show lower hardness values due to the loss of mineral from intertubular dentin. Interestingly, during the caries-forming process, the tubules become obliterated with whitlockites, thus reducing permeability of the caries-affected dentin.[15]

In general, SEM analysis revealed a thicker hybrid layer formation for the Single Bond groups compared to the Adper Prompt L Pop groups. This is most likely due to the specific feature of each adhesive system, one being an "etch and rinse" system that uses an aggressive phosphoric acid etching and the other a self-etching adhesive that is less aggressive. The hybrid layers of both Single Bond and Adper Prompt L Pop for the caries-affected dentin were thicker than those formed in normal dentin, an observation which was also previously reported.[9,15] This phenomenon may be due to the partially demineralized nature of intertubular dentin in caries-affected dentin, which permits deeper acid demineralization and deeper monomer infiltration compared to normal dentin.

CONCLUSIONS

Within the limitations of this study, the authors have failed to reject the null hypothesis, since bond strengths of the "1-step" self-etching adhesive were significantly higher to normal dentin compared to caries-affected dentin.

Further studies are still necessary to uncover the mechanism of adhesion between adhesive systems and caries-affected dentin and the long-term bonding durability to this type of substrate.

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