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Effect of Different Bonding Conditions on the Shear Bond Strength of Two Compomers to Bovine Dentin

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Abstract

Aim: Despite the improvements to compomer materials, the bond strength of these materials remains inferior to "composite/resin bonding" systems and limits their clinical use. The purpose of this study was to evaluate the effect of acidic conditioning with phosphoric acid and Prompt L-Pop (PLP) on the shear bond strength of two compomers Dyract AP (DAP) and Composan Glass (CG) to dentin.

Methods and Materials: Sixty extracted bovine teeth were used to test the shear bond strength of two compomers to flat dentin labial surfaces. The dentin specimens were randomly assigned to six groups of ten specimens each: Group 1: DAP and Prime & Bond NT (PBNT); Group 2: DAP/PBNT with a 15 second dentin etch prior to bonding; Group 3: DAP placed with PLP adhesive; Group 4: CG and Compobond NE (CBNE); Group 5: CG/CBNE with a 15 second dentin etch; and Group 6: CG placed with PLP adhesive. The specimens were stored at 37ºC with 100% humidity for 24 hours then mounted and sheared using an Instron Universal Testing Machine at a cross head speed of 0.5 mm/min. The results were recorded in Mega Pascals (MPa). The sheared specimens were examined under a light microscope, and the type of failure (adhesive, cohesive, or mixed) was recorded.

Results: The mean dentin shear bond strength value (MPa) for the groups was: Group 1 (11.6±3.9); Group 2 (13.2±3.3); Group 3 (12.4±2.0); Group 4 (13.0±4.3); Group 5 (19.3±3.7); and Group 6 (13.1±3.0). One way analysis of variance (ANOVA) and Tukey HSD post-hoc tests detected a significantly higher bond strength ($P \leq 0.003$) for group 5. For groups 1, 3, 4, and 6, the mode of failure was mostly adhesive. When acid etching of dentin was performed (groups 2 and 5), cohesive fracture within dentin was the predominant mode of failure. Acid etching and the use of PLP significantly reduced the number of adhesive fractures and reduced variability in the shear bond strength results.

Conclusions: Acid etching significantly increased the shear bond strength of CG to dentin but did not affect DAP. The application of PLP resulted in a shear bond strength not statistically different from PBNT or CPNE. CG bond to dentin is improved with acid etching using phosphoric acid. However, PLP provided no significant improvement in the shear bond strength of DAP and CG.

Keywords: Dentin adhesion, compomer, self-etching primers, dentin etching, Prompt L-Pop

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Introduction

Bonding to enamel via the acid etch technique is a well-established technique, and it is supported by numerous *in vitro* and *in vivo* studies. Enamel etching is accomplished using 30-40% phosphoric acid with the resulting surface characterized by abundant microporosities which are readily penetrated by a low viscosity resin to form resin tags providing micromechanical retention.[1,2] Compared to enamel bonding dentin is far more challenging due to some inherent characteristics of dentin which complicate bonding. These characteristics include variable tubular structure, high organic content, and positive fluid flow.[3-6]

Adhesion to dentin is primarily micromechanical. The three step procedure consists of brief acidic conditioning with phosphoric acid followed by the application of a hydrophilic primer carried in a solvent. Water chasing solvents such as ethanol or acetone are commonly utilized to facilitate penetration of the monomers into the exposed network of collagen fibrils. The resulting structure consists of entangled collagen fibrils infiltrated with polymerized resin called the hybrid layer.[4,7,8]

The simultaneous etching of enamel and dentin, or total etch technique, and the developments made in chemical adhesives have improved the bond strength and reduced microleakage of resin restoratives.[9-11] Current developments have focused on simplifying the application of bonding agents by decreasing the time and steps required for placement. As a result, manufacturers have combined the primer and adhesive into a single component but have still maintained separate etch and rinse steps. This method of bonding is commonly called "two-step bonding."[1]

Acid-etching of dentin to remove the smear layer and demineralize the tooth surface is the standard surface treatment before bonding of resin based composites to dentin. However, the exposed denatured collagen fibrils easily collapse during air drying preventing infiltration with the resin monomers. To prevent the collapse of the collagen network, the dentin should be kept moist to maintain the interfibular space.[12] A practical problem in this approach is to determine the ideal level of moisture needed. Another approach to prevent the collapse of the collagen network is to leave the smear layer in place while using acidic monomers to etch through the smear layer into the underlying dentin and avoid rinsing and drying the conditioned surface.[12] This newer approach to dentin bonding is called "the self-etching technique."

Self-etching primers condition and prime the enamel and dentin surfaces without rinsing. Etching dentin partially removes the smear layer and opens dentinal tubules, however, the mild acidity of self-etching primers does not completely remove the smear layer leaving the tubules plugged with smear debris. This partial dissolution of the smear layer results in a hybridized zone of about 2 μm thick which contains some entrapped materials.[13-15] Tay et al.[16] studied the effect of self-etching primer acidity and smear layer thickness on the bond strength and reported a minimum pH value of 2.8 is required for self-etching primers to penetrate beyond the smear layer and etch the underlying mineralized dentin and form a hybrid layer.

Despite the improvements of compomer materials, the dentin bond strength of these materials remains inferior to “composite/resin bonding” systems. Dentin bond strength should be approximately 20 MPa for good clinical bonding. However, current compomers exhibit only 50-60% of this degree of bond strength. Since compomers are closer in their chemistry to composites than glass ionomer cements it is quite possible their bond strengths to dentin could benefit from the acid etching process.

Van Meerbeek et al.[1] classified Prime & Bond NT (PBNT) (Dentsply, Konstanz, Germany) as a two-step etch and rinse adhesive that can be used with both composites and polyacid modified composites. Although numerous studies[17-21] showed acid etching of enamel prior to bonding with PBNT significantly increases the bond strength to Dyract AP (DAP) (Dentsply, Konstanz, Germany), The manufacturer’s instructions recommend acid etching of enamel only when enamel beveling was performed or when maximum adhesion is required.

Several one-step all-in-one adhesive systems have been developed. One such system is Prompt L-Pop (PLP) (ESPE, Seefeld, Germany). This product is a strong self-etching primer containing methacrylated phosphoric acid esters with a pH <1 which is much lower than all other self-etching primers and it is recommended for use with both composites and compomers.[1]

The purpose of this study was to evaluate the effect of acidic conditioning with phosphoric acid and PLP on the shear bond strength of two compomers: DAP and Composan Glass (CG) (Promedica, Neamanster, Germany) to dentin.

Methods and Materials

The materials used in this study were DAP which is a widely used extensively investigated compomer material and CG which is a compomer material widely used in Europe although less frequently investigated. Both materials are acetone-based and do not require separate conditioning steps. The compositions of the adhesives investigated are shown on Table 1.

Sixty freshly extracted bovine incisors were cleaned and kept refrigerated in a 0.02% solution of distilled water and thymol. The crowns were sectioned using a diamond disc, and the teeth were embedded in Teflon® molds filled with self-cure acrylic resin. The labial surfaces were flattened using 240 and 400 grit silicone carbide paper under a water coolant. Dentin surfaces at least 5 mm in diameter were exposed, and care was taken to expose only superficial dentin.

The mounted teeth were randomly distributed into six groups. Immediately before bonding the dentin surfaces were freshened using 600 grit silicone carbide paper. A split Teflon® mold, 5 mm in diameter and 3 mm long, was placed over each tooth perpendicular to the polished

Table 1. Composition and lot number of the adhesive systems used in the study.

Product Name	Adhesive Components	Lot #
Dyract AP	PENTA, UDMA, other	0307000824
Prime & Bond NT	resin (R5-61-1, T-resin, D-resin), nanofillers, initiators, stabilizers, cetylamine hydroflouride, acetone	0306001018
Composan Glass Compobond NE	BIS-GMA, UDMA, HEMA, BHT, acetone, organic acids	95587 24854
ADPER Prompt L-Pop	Water, methacrylated phosphoric, acid esters, fluoride complex, photoinitiator (BAPO), stabilizer, parabenes.	175425

Table 2. Adhesive systems tested and their application protocol.

Group	Compomer	Adhesive	Acid Etching	Protocol
1	Dyract AP	Prime & Bond NT	None	**Prime & Bond NT** Apply on dentin for 20 seconds, remove solvent by air 5 seconds, light cure 20 seconds. **Acid Etch** Apply phosphoric acid 15 seconds, wash 30 seconds, evacuate excess moisture then use damp cotton pellet. **Prompt L-Pop** Activate, apply on dentin, rub surface with moderate pressure using air, cure for 10 seconds. **Compobond NE** Dispense and mix for 5 seconds apply and scrub for 30 seconds dry for 5 seconds, polymerize for 20 seconds. Apply a second layer for 5 seconds. Do not polymerize.
2	Dyract AP	Prime & Bond NT	Yes	
3	Dyract Ap	Prompt L-pop	None	
4	Composan Glass	Compobond NE	None	
5	Composan Glass	Compobond NE	Yes	
6	Composan Glass	Prompt L-pop	None	

surface. A metal ring was used to secure the mold and the appropriate material was placed according to the manufacturers instructions.

To ensure a moist bonding technique the residual water from the etchant-rinsing step was removed by blotting the surface with a moist cotton pellet so the resulting surface was visibly moist without excess water.[7] The six groups were treated as shown in Table 2.

All specimens were light cured using a Elipar Highlight (3M, ESPE, St. Paul, MN, USA). The intensity of the light was monitored periodically with a radiometer (Demetron/Kerr, Danburg, CT, USA) to ensure 400 mW/cm^2 was exceeded. For all specimens, the curing light was held 2 mm away from the restoration and each layer was cured for 40 seconds.

The specimens were stored in distilled water at 37°C for 24 hours. They were then mounted with the treated surfaces parallel to the shearing rod of the Instron Universal Testing Machine (Instron Corporation, Canton, MA, USA), sheared to failure at a cross head speed of 0.5 mm/min, and the results recorded in Mega Pascals (MPa). The testing was carried out at room temperature of 23°C and relative humidity of 50%.

A one way analysis of variance (ANOVA) was used to detect any significant differences ($p \leq 0.05$) in bond strengths among the groups. Post hoc comparisons were made using the Tukey HSD test.

The failed surfaces were examined under a light microscope (Traveling Mic., By TITAN Measuring Microscope, Buffalo, NY, USA) at a magnification of x10, and the mode of failure of the specimens were recorded according to the following categories:

- Adhesive failure at the dentin – restoration interface (no compomer on dentin surface).
- Cohesive failure in the dentin if some of dentin remained on the compomer.
- Cohesive failure in the compomer if remnants of the compomer remained on dentin.
- Mixed failure in the dentin and compomer.

The results were subjected to chi-square analysis using SPSS for Windows 2000 version.

Results

Shear Bond Strength

Mean shear bond strengths to dentin for the six treatment groups are shown in Table 3. One way ANOVA showed a significant difference between the three CG groups *($p \leq 0.001$)*. Tukey HSD post-hoc test indicated group 5 (CG placed after acid etching of dentin) had significantly higher bond strength to dentin *($p \leq 0.003$)* than all of the other groups. Groups 1, 2, 3, 4, and 6 had bond strengths ranging between 11.6 MPa to 13.2 MPa. DAP bond strength to dentin was not affected by the variations in the application techniques tested in this study.

Failure Mode Analysis

The mode of failure for the six bonding test groups, as determined by observation under optical microscope, is shown in Table 4. For groups 1, 3, 4, and 6, the mode of failure was mostly adhesive in nature. When acid etching of dentin was performed, the mode of failure changed from predominantly adhesive in nature to cohesive fracture within dentin.

Although the results of chi-square analysis were highly significant *($p<0.00$)*, inference could not be made because of the small sample size and 75% of the cells have an expected count less than five.

Discussion

The dental samples were carefully prepared to ensure only the outer dentin was exposed for bonding. Bonding to deeper dentin is complicated by its more heterogeneous structure, variable tubular density, and tubular fluid flow. Therefore, bonding to inner dentin may add more variables that could interfere with the ability to evaluate the two approaches.[5,6]

Table 3. Mean bond strength and standard deviation for the test groups.

Group	Description	N	Mean, MPa (SD)	Coefficient Variation
1	Dyract AP	10	11.6(3.9)[a]	33%
2	Dyract AP/Etch	10	13.2(3.3)[a]	25%
3	Dyract AP/L-pop	10	12.4(2.0)[a]	16%
4	Compoglass	10	13.0(4.3)[a]	33%
5	Compoglass/Etch	10	19.3(3.7)[b]	19%
6	Compoglass/L-pop	10	13.1(3.0)[a]	23%

Note: Groups identified with different superscript letters are significantly different *($p < 0.05$)*

Table 4. Failure modes of test groups.

Group	Description	Failure			
		Adhesive	Cohesive (in dentin)	Cohesive (in compomer)	Mixed
1	Dyract AP	90%	10%		
2	Dyract AP/Etch	40%	40%		20%
3	Dyract AP/L-Pop	80%	10%	10%	
4	Compoglass	100%			
5	Compoglass/Etch	20%	80%		
6	Composan/L-Pop	80%			20%

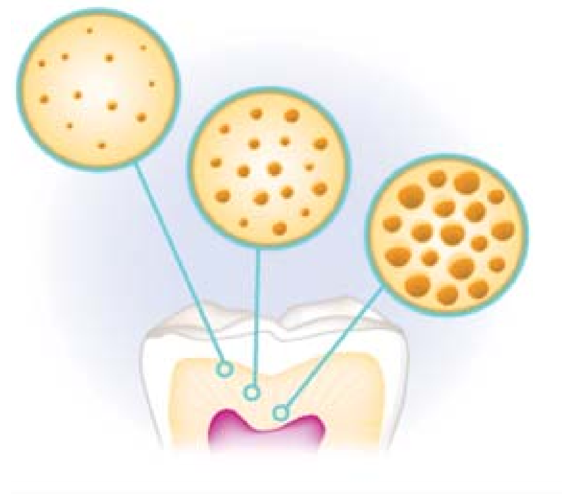
It has been clearly demonstrated a separate acid etching step prior to applying an etch and rinse adhesive is indispensable for reliable enamel and dentin bonds to composites.[20] However, this step seems to be omitted or optional for most compomer products presently available. This trend is surprising given the inferior bond strengths of compomers, and the chemistry and the bonding systems for compomers are similar to composites. Furthermore, the bonding mechanisms of compomer involve hybrid layer formation like bonded resin-based composites.

Although the effect of enamel acid etching on the bond strengths of some compomers have been reported in the literature, dentinal acid etching before bonding of some of the newer versions of compomers has not been investigated sufficiently. Furthermore, a direct comparison between phosphoric acid etching and the use of strong self-etching adhesives to determine which of the two approaches warrants further study is indicated. All the three adhesives studied here are universal adhesives marketed for use with compomers and composites.

The effect of phosphoric acid pre-conditioning on the bond strength of DAP to dentin is a controversial issue in the literature. Studies using the older versions of Prime & Bond showed improved bonding.[23,24] However, studies of the newer version, PBNT, show conflicting results.[15,22] In the present study bond strength to dentin was significantly increased when dentin etching was performed only with CG (group 5). DAP bond strength was not significantly affected by acid etching of dentinal surface which is in agreement with the results of Sunico et al.[22]

PBNT is an acetone based solution of phosphoric acid esters containing PENTA monomer which possesses acidic properties with a pH of 2.2 which is lower than the minimum value 2.8 reported by Tay et al.[16] Therefore, PBNT may present mild self-etching characteristics when applied to dentin with intact smear layer and produce bond strength values similar to acid etched dentin.[1,16]

CG bond strength to dentin increased significantly after acid etching of dentin, and it was significantly higher than all the other groups. This is probably due to the different



composition of the primer. Compobond NE (CBNE) (PROMEDICA, Neumünster, Germany) contains hydroxyethylmetha-acrylate HEMA which is a water soluble primer. Organic acids are also added to the primer which might impart some self-etching properties to the material allowing it to have improved adhesion to enamel and dentin.[25]

Eick et al.[26] hypothesized self-etching acidic primer, when used to demineralize the smear layer, may leave a collagen residue which becomes a part of the hybrid layer and may affect adhesion. Furthermore, according to Gordon et al.,[27] as the acidic primer demineralizes the dentin surface, the concentration calcium phosphate increases. This neutralizes the primer and limits the depth of etching, thus, affecting adhesion.[27] The results of this study cannot be explained by this hypothesis since DAP values were not significantly different with or without acid etching.

Several authors[28-31] have pointed out the collagen demineralized layer may play a significant qualitative role in the ultimate bond strength to dentin, but its quantitative role may be less significant. Gwinett[32] showed the total bond strength to resin based materials to dentin is due to any or all of the following: resin tag formation, hybrid layer formation, and surface adhesion. Hypothetically, it is quite possible surface adhesion might have played a role in this case and contributed to the final bond strength. However, McLean[33] stated there is still little evidence compomers can adhere to dentin by chemical bonding and, thus, conventional acid etching is still required to obtain high bond strengths.

It is interesting to note acid etching with phosphoric acid might not always significantly increase the bond strength to dentin. However, it will always significantly reduce the percentage of purely adhesive fractures at the dentin-compomer interphase and increase the percentage of cohesive fracture in the dentin or compomer. Analysis of the failure mode data (Table 4) indicates the actual bond strength of acid etched specimens might be higher than the measured values since the dentin failed cohesively before the bonded surfaces actually failed.

In this study the use of PLP with the two compomers used produced bond strengths that are not statistically different from those of PBNT or CBNE. This result is in agreement with some previous studies.[34,35]

According to Watanabe et al.[36] self-etching primers create diffusion channels into intact calcium-rich dentin. This prevents the loss of dentin mass but solubilizes enough apatite crystals from around collagen fibrils to permit infiltration of adhesive monomers. Therefore, hybridization created by self-etching primers is free from defects and is continuous from resin to calcium rich dentin.[36] Furthermore, Perdigao et al.[37] believed the bonding mechanism provided by self-etching primers may be more stable with time because collagen fibers are surrounded by hydroxyapatite crystals which might protect it against hydrolysis and early degradation of the bond.

The two materials tested showed standard deviations of approximately 30% around the mean which is to be expected considering the heterogeneousity of the dentin surface. The use of PLP and phosphoric acid to precondition the dentin surface produced a coefficient of variation of the measured shear bond strength which is about 20%, (i.e., 30% less) as shown in Table 4. This tendency is probably due to the reduced number of surface voids or defects, and it could possibly indicate a more reliable bond between the dentin and the compomers used.

Conclusion

Based on the results of this study there appears to be strong evidence acid etching of dentin with phosphoric acid could significantly improve the bond with compomer materials. The interaction between the type of adhesive system and the surface conditioning used is material specific, and clinicians should be aware of these effects to be able to optimize the performance of the materials they use. Further studies are needed to test other compomer/adhesive systems and to investigate the effect of cavity depth on the bond.

To summarize, the present study found the following:

1. Acid etching significantly ($p \leq 0.003$) increased the bond strength of CG to dentin but did not affect DAP.
2. The application of PLP resulted in bond strengths not statistically different from those of PBNT or CBNE.

CG bond to dentin is improved with acid etching using phosphoric acid. However, PLP provided no significant improvement in the shear bond strength of DAP and CG.

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