# An in vitro comparison of acid etched vs. nonacid etched dentin bonding agents/composite interfaces over primary dentin

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## Abstract

*The purpose of this study was to evaluate acid etchant penetration on dentin bonding agents and its effect on the composite resin bond strength. Forty primary molars were mounted, then the buccal and lingual surfaces were prepared into dentin. The teeth were divided into four groups of 10, and four dentin bonding agents were placed on the buccal and lingual surfaces of exposed dentin, as recommended by the manufacturers. One surface of each tooth was etched randomly for 60 sec with 35% phosphoric acid. A standardized tube of composite resin was placed on each dentin surface and polymerized for 60 sec. The tubes were sheared off with an Instron® Testing Machine. The specimens then were sectioned to be examined by a scanning electron microscope (SEM). Results demonstrated shear strengths (kg/cm²) of etched (e) and unetched (u) dentin bonding agents to be: Scotchbond® (3M Dental Products, St. Paul, MN) (e) 116.7 ± 37.7, (u) 116.7 ± 63.0; Scotchbond 2® (3M Dental Products, St. Paul, MN) (e) 112.0 ± 40.6, (u) 127.0 ± 38.7; Gluma® (Bayer Dental, Leverkusen, Federal Republic of Germany) (e) 80.1 ± 21.7, (u) 107.0 ± 16.6; Bondlite® (Kerr Manufacturing Co., Romulus, MI) (e) 53.4 ± 34.7, (u) 79.1 ± 26.3. The analysis of variance (ANOVA) demonstrated a statistical significance in variance at the $P < 0.001$ level. Scheffe's Test indicated no statistically significant differences between the bond strengths of etched vs. nonetched dentin bonding agents and composite resin. SEM evaluation indicated that the acid etchant penetrated none of the dentin bonding agents. (Pediatr Dent 13:204–07, 1991)*


## Introduction

The increased use of polymerized composite resin in the last decade has stimulated much research to help achieve a superior restoration through better materials and methods. Buonocore's introduction of a method of acid etching enamel in 1955 opened a new realm of restorative dentistry. This procedure, due to the high inorganic content of enamel, improved restoration marginal seal and added micromechanical bond strength.

The search for a bonding agent for dentin began; however, this proved more of a challenge as the organic component of this tissue contributed to low bond strengths. Pursuing the goal of increasing bond strengths, Bowen (1965) proposed a dentin bonding agent (NPG-GMA). Because of the random pattern of hydroxyapatite crystals in dentin and the low tensile strength of collagen, it was obvious that the micromechanical bond achieved in enamel bonding was not possible with dentin bonding; a chemical bond was necessary (Bowen et al. 1984). Another contributing factor was the presence of a "smear layer" on dentin. Operative procedures on dentin produce a layer of loosely bound debris, caused by frictional heat and deformation from cutting procedures. This also contributed to a low bond strength to dentin. Removing the smear layer has been found to increase the effectiveness of the dentin bonding agent (Bowen et al. 1984).

Several generations of dentin bonding agents have been developed since the first ones were introduced. The second generation of these agents, known as dentin/enamel bonding agents, basically falls into two groups. The first was halophosphorous esters of Bis-GMA, which relied on a phosphate-calcium bond. The second group was categorized as polyurethanes (Setcos 1988). These bonding agents were halophosphorous esters of hydroxyethyl methacrylate (HEMA) which relied on a phosphate-calcium bond. The newest generation of dentin bonding agents include primer components which treat or alter the dentin surface, as well as its smear layer, before bonding. This results in the creation of a mechanical bond by the infiltration of monomers into a zone of demineralized dentin, where the monomers polymerize and interlock with the dentin matrix (Erickson 1989). These new dentin bonding agents have shown bond strengths comparable to those of etched enamel and glass ionomer materials (Chan et al. 1985; Erickson 1989).

The etching of enamel is essential in providing a successful composite resin restoration; however, if the acid etchant is placed inadvertently on dentin, damage to the pulpal tissue can occur (Stanley et al. 1975; Macko et al. 1978). Etching dentin in vivo has shown a fivefold increase in dentin permeability and an increased penetration of bacteria (Pashley et al. 1983; Meryon and Jakeman 1985). Fusayama (1987) on the other hand, has recommended phosphoric acid etching of vital dentin before placing dentin bonding agents. He believes that dentin sensitivity is secondary to inadequate bonding. The dentinal tubules are opened following etching, yet polymerization shrinkage stresses pull the bonding agent away from the dentin surface. The lack of adaptation of the bonding agent to the dentin creates a bond deficiency, allowing bacterial invasion and sensitivity. Although Fusayama advocates phosphoric acid etching of dentin, the accepted standard of care is in opposition to this recommendation. The protection of dentin during etching is necessary. Could placing a dentin bonding agent before acid etching the enamel protect the dentin and result in a bond strength comparable to that of a dentin bonding agent not subjected to acid etching?

Chan and Jensen (1986) found, in an in vitro study, that treating the dentin surface with dentin bonding agents effectively could reduce acid penetration. However, Eick and Welch (1986) concluded that chlorophosphate ester-type adhesives were penetrated under exposure to phosphoric acid. They found the polyurethane type of bonding agents were effective in preventing acid from etching completely through the bonding agent.

The recommendation that glass ionomers be used as liners to protect the dentin from acid etching has been well received (Wilson and Kent 1971). These materials provide fluoride release, biocompatibility and bonding to unetched dentin. Placing the glass ionomer liner, however, is time consuming and technique sensitive (Phillips and Bishop 1985). The purpose of this in vitro study was to ascertain the ability of dentin bonding agents to protect the dentin during enamel acid etching, without compromising bond strength. A positive evaluation may allow the clinician to eliminate the routine placement of a liner, thereby decreasing chair time and possibly increasing bond strength.

## Materials and Methods

Forty extracted or exfoliated primary teeth were obtained for this in vitro study. The teeth were stored in a 0.1% thymol solution until the experimental procedure began, thereby preventing dehydration. The teeth were mounted in 2.5 cm retention tubes held fast by acrylic.

Each tooth was sectioned on the buccal and lingual surfaces. The sections, prepared with a high-speed diamond underwater spray, exposed a minimum of 5 $mm^2$ of dentin. The teeth were divided into four groups of 10, each of which had a commercially available dentin bonding agent placed on the buccal and lingual sections according to the manufacturer's instructions. The groups are as follows: Group I — Scotchbond® (3M Dental Products, St. Paul, MN); Group II — Scotchbond 2® (3M Dental Products, St. Paul, MN); Group III — Gluma® (Bayer Dental, Leverkusen, Federal Republic of Germany); Group IV — Bondlite® (Kerr Manufacturing Co., Romulus, MI). Randomly, the buccal and lingual section on each tooth was acid etched for 60 sec with 35% phosphoric acid gel. Each exposed dentinal surface then had a standardized core of light-cured composite resin bonded to the dentin bonding agent and polymerized for 60 sec. Each core, containing composite resin within clear plastic tubing, was 5 mm in diameter and extended 10 mm from the dentinal surface.

Following placement of the composite, each core was placed under a pressure rod, extending from an Instron® (Instron Engineering Corp., Canton, MA) testing machine. A shear force was applied at the bond site. A comparison of the shear strengths of acid etched dentin bonding agents to the composite cores was made to the shear strengths of nonacid etched dentin bonding agents to composite cores, using a randomized block design.

After the test for shear bond strength, the specimens were sectioned and examined with an SEM. Photomicrographs were made to evaluate acid penetration of the dentin bonding agents on both the etched and unetched sections obtained from the area adjacent to where the composite core had been placed.

## Results

Results demonstrated shear strengths (kg/cm2) of etched (e) and unetched (u) dentin bonding agents to be: Scotchbond (e) 116.7 ± 37.7, (u) 116.7 ± 63.0; Scotchbond 2 (e) 112.0 ± 40.6; (u) 127.0 ± 38.7; Gluma (e) 80.1 ± 21.7, (u) 107.0 ± 16.6; and Bondlite (e) 53.4 ± 34.7 (u) 79.1 ± 26.3 (Fig 1, see next page). The analysis of variance (ANOVA) demonstrated a statistical significance in variance at the $P < 0.001$ level. Scheffe's Test indicated no statistically significant difference between the bond strengths of etched vs. nonetched dentin bonding agents and composite resin. SEM evaluation indicated that the acid etchant penetrated none of the dentin bonding agents.

## Discussion

Four different commercially available dentin bonding agents were evaluated, comparing shear strengths of the etched and unetched adhesives to composite. There were no statistically significant differences between the etched and unetched sections. The mean shear bond strengths were greater than those reported in another study comparing similar dentin bonding agents to primary dentin (Fagan et al. 1986). This same study demonstrated no significant differences between

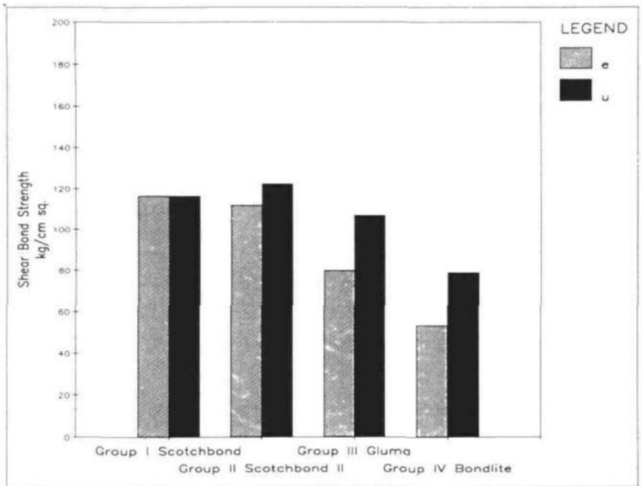


**Fig 1.** Bar graph illustrating the mean shear bond strength (kg/cm²) of the experimental bonding agents.

dentin bond strengths in primary and permanent teeth. The mean shear values obtained in our study were similar to mean values reported by other investigators for shear strengths of dentin bonding agents to permanent dentin (Reinhardt et al. 1987). These values give us a baseline from which to evaluate dentin bonding agents in vitro. A clinical situation will determine the true success or failure.

The mean shear bond strengths demonstrated a significance in variance ($P < 0.001$). Although there was a significant variance, when the samples were compared to each other there was no significant difference. A randomized block design was the format used for specific comparisons with the Scheffe's Test. The data must be evaluated carefully. The wide variation in bond strengths within each group is indicated by the large standard deviation for each group. Dentin bonding agents have less predictable bond strengths to dentin than to enamel. This study demonstrates that data appearance can be misleading, and appropriate comparison must be made after a significance in variance is seen.

SEM evaluation of all four commercially available dentin bonding agents used in this study revealed no penetration of the dentin bonding agent by the acid etchant. The results have demonstrated that the placement of dentin bonding agents protected the primary dentin from acid penetration during phosphoric acid etching. This certainly could have an effect on the routine restorative dentistry practice. Following tooth preparation, a dentin bonding agent may be placed over the dentin and polymerized. A bevel then can be placed on all enamel margins, removing any bonding agent that was present on the enamel margins, and the enamel may be acid etched.

The results of this study also indicated that if any phosphoric acid inadvertently contacts the previously placed dentin bonding agent, the bond strength to composite resin will not be reduced significantly. It is important to note that no additional bonding agent was placed following the etching of the bonding agent. A thin layer of bonding agent could be placed easily following acid etching, since the etched enamel must have an unfilled resin placed before filled composite resin adaptation and may provide improved bond strength.

When dentin bonding agents are placed on a prepared surface and polymerized, the ruffled nature of the film caused by polymerization shrinkage is evident. Fig 2 (250x) shows the ruffled nature of the dentin bonding agents. Fig 3 (250x) shows the surface of bonding resins after they have been etched for 60 sec. The

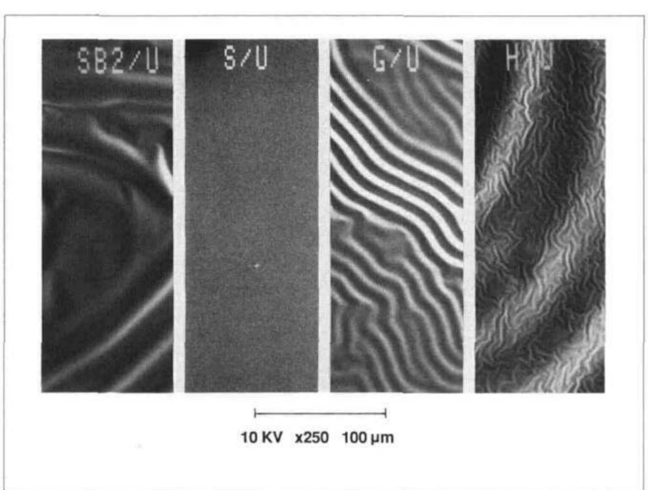


**Fig 2.** Surface structure of Scotchbond 2 (SB2). Scotchbond (S), Gluma (G), and Bondlite (H) 250x.

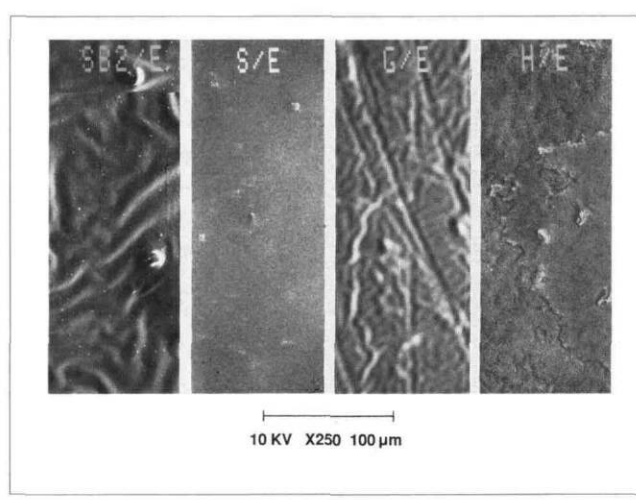


**Fig 3.** Surface structure of the dentin bonding agents (250x) following a 60 sec etch.

dentin adhesive surfaces are altered slightly yet remain intact with no evidence of exposed dentinal tubules.

Scotchbond and Bondlite are second generation chlorophosphate esters. Scotchbond 2 and Gluma are third generation dentin bonding agents, involving more than one application step. Gluma has a cleanser, primer and resin, while Scotchbond 2 has a primer followed by a bonding resin. Both of these systems remove or alter the smear layer of dentin to help create a mechanical bond. Eick and Welch (1986) found only the polyurethane adhesives were effective in maintaining a dentinal seal when acid was applied; the chlorophosphate ester types were not. It also was suggested that a thicker film may have been more resistant to acid dissolution. Since no acid penetration occurred in this study, it is possible that placement of a thicker layer was achieved, resulting in more resistance to the acid etchant. Aggressive thinning, spreading, or drying of bonding agents with an air syringe should be avoided during adhesive placement.

Presently, placing calcium hydroxide or glass ionomer cement is recommended to protect the dentin during acid etching. Placing a dentin bonding agent may prove to decrease operative chair time, while maintaining excellence in restorative dentistry. A dentin bonding agent is advantageous compared to a calcium hydroxide liner, which does not allow for a bond to dentin and presents the risk of hydrolysis when placed under composite resin restorations (Donly et al. 1990).

Further investigation is necessary in several areas. First, biocompatibility of resins has been questioned. Preparations that extend beyond an ideal depth may require placing a protective base before the bonding agent is applied. The hygroscopic properties of resins also present a concern. Possible hydrolysis of the unfilled dentin bonding resin could create restoration defects. These concerns should be addressed.

## Conclusion

The shear bond strengths of dentin bonding agents to primary dentin were not affected significantly by the etching process. This study demonstrated that dentin bonding agents were effective in protecting primary dentin during enamel etching.

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