Parameters that Affect *in vitro* Bonding of Glass-ionomer Liners to Dentin

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The purpose of this study was to evaluate the effects of two concentrations of poly (acrylic acid) (10 and 25%), three treatments (untreated, passive conditioning, and active conditioning), and two storage conditions (24 hours in 37°C water and thermal cycling) on the in vitro *tensile bond strength of three commercial glass-ionomer liners to human dentin. Bond strengths to untreated dentin after storage for 24 hours ranged from 19.0 to 21.7 kg/cm² for Glasionomer Base Cement, Cement/Liner, and Ketac-Bond, but dropped to a range of 4.9 to 9.7 kg/cm² after thermal cycling. Active conditioning with 10% acid resulted in bond strengths after 24-hour storage that ranged from 23.5 to 44.0 kg/cm², compared with values from 21.7 to 38.0 kg/cm² with active conditioning using 25% acid. Active conditioning with 10% acid resulted in bond strengths after thermal cycling that were in the range of 15.8 to 27.4 kg/cm² and were 80 to 320 percent higher than values resulting from passive conditioning under these conditions. Active conditioning with 10% acid for 30 seconds produced a bond strength for Glasionomer Base Cement of 44.0 kg/cm², compared with a bond strength of 28.7 kg/cm² for a 10-second active conditioning. Qualitative analysis of scanning electron photomicrographs showed that dentin tubules were opened to a greater extent by active conditioning with 25% acid than by passive conditioning with 10% acid.*

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Introduction.

Glass-ionomer cements have shown evidence of chemical bonding to tooth structure (Coury *et al.*, 1982; Maldonado *et al.*, 1978; Wilson *et al.*, 1983). Several studies have shown that the removal of the smear layer from dentin decreased microleakage around the restoration (Hinoura *et al.*, 1986; Jodaikin and Austin, 1981; Pashley and Depew, 1986; and Powis *et al.*, 1982). The use of poly (acrylic acid) has been shown to remove the smear layer and to increase the bond strength of polyacid cements to dentin without increasing pulpal sensitivity (Barakat and Powers, 1986; Hinoura *et al.*, 1986). Continuous brushing of 35% phosphoric acid on enamel resulted in a more debris-free surface than an unbrushed one (Baharav *et al.*, 1987).

The purpose of this study was to evaluate the effects of two concentrations of poly (acrylic acid), three treatments (untreated, passive conditioning, and active conditioning), and two storage conditions on the *in vitro* tensile bond strength of three commercial glass-ionomer liners to human dentin.

Materials and methods.

Products, batch numbers, and manufacturers of the glass-ionomer liners and the conditioners studied are listed in Table 1. The powder-liquid ratios (g/g) used for the liners were: Ketac-Bond, 3.4/1; Cement/Liner, 1.25/1; and Glasionomer Base Cement, 2.6/1.

Freshly extracted, non-carious human teeth were embedded in resin with the buccal or lingual surface exposed. Flat dentinal surfaces were prepared by being polished with wet silicon carbide paper to a finish of 600 grit. The teeth were stored in distilled water until use and were not re-used.

The dentin surfaces were prepared according to the following conditions: untreated, passive conditioning with 10% acid; active conditioning with 10% acid; passive conditioning with 25% acid; and active conditioning with 25% acid. In the passive-conditioning technique, a drop of acid was applied to the dentin surface for 30 seconds without rubbing. In the active-conditioning technique, the acid was applied to the dentin surface for 30 seconds by continuous rubbing with a small sponge. For Glasionomer Base Cement, these conditions were repeated with a 10-second application of acid. After conditioning, the dentin surfaces were rinsed for 10 seconds and blown dry with air for 10 seconds. Scanning electron photomicrographs of dentin before bonding were obtained for representative samples for each of the four 30-second conditioning treatments.

The glass-ionomer liners were mixed according to manufacturer's instructions and applied to an area of dentin (5 mm in diameter) limited by adhesive tape. The nominal area of bonding was 19.6 mm².

The mold used to prepare the truncated-cone tensile-bond-strength samples and the assembly used for debonding have been described before (Barakat and Powers, 1986; Watanabe *et al.*, 1988). Before being tested, half of the samples were stored in water at 37°C for 24 hours, and the other half were subjected to thermal cycling in water baths at 5 and 55°C for three minutes *per* bath and 100 cycles *per* bath (Model CHCB/2050A, Standard Environmental Systems, Inc., Totowa, NJ). Samples were loaded in tension at a cross-head speed of 0.1 mm/minute on a testing machine (Model TT-BM, Instron Corp., Canton, MA). The tensile bond strength was calculated as the load at failure divided by the nominal area of the liner. The locations of failure were noted.

Five specimens were evaluated for each of the experimental combinations. Means and standard deviations were calculated. Data were analyzed by analysis of variance with a factorial design (Dalby, 1968). Means were compared with a Tukey interval calculated at the 95% level of confidence (Guenther, 1964). Differences between two means that were larger than the Tukey interval were statistically significant.

TABLE 1
CODES, PRODUCTS, BATCH NUMBERS, AND MANUFACTURERS OF GLASS-IONOMER LINERS AND CONDITIONERS TESTED

Product	Batch Number	Manufacturer
Glass-ionomer liners:		
Ketac-Bond	P 0012	ESPE-Premier Sales Corp. Norristown, PA 19401
Cement/Liner	P 068764 L 0587172	Parkell Products Farmingdale, NY 11735
Glasionomer Base Cement	P 058701 L 128701	Shofu Dental Corp. Menlo Park, CA 94025
Conditioners		
10% TGH-10	—	Ho Dental Co., Inc.
25% TGH-25	—	Goleta, CA 93117

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TABLE 2
IN VITRO BOND STRENGTH (kg/cm²) OF GLASS-IONOMER LINERS TO DENTIN UNDER DIFFERENT CONDITIONS

	Liners			
Condition	Ketac-Bond	Cement/Liner	Glasionomer Base Cement	Glasionomer Base Cement (10s)
24 hours-37°C water				
Untreated	21.7 (6.2)*	20.5 (5.1)	19.0 (4.3)	—
10% passive	24.1 (4.0)	19.6 (5.0)	34.1 (5.4)	21.6 (4.6)+
10% active	23.5 (6.7)	25.7 (3.0)	44.0 (4.3)	28.7 (3.1)
25% passive	18.0 (5.0)	24.6 (3.3)	34.3 (3.1)	22.5 (3.6)
25% active	21.7 (4.4)	22.2 (4.5)	38.0 (0.8)	41.1 (1.8)
Thermocycled				
Untreated	4.9 (1.5)	9.7 (2.2)	6.3 (1.8)	—
10% passive	10.4 (3.2)	8.5 (1.3)	6.6 (2.3)	—
10% active	19.1 (3.0)	15.8 (3.7)	27.4 (6.0)	—
25% passive	7.6 (3.0)	26.1 (2.1)	16.8 (3.8)	—
25% active	14.5 (4.4)	25.5 (1.9)	20.9 (6.1)	—

*Mean value of five replications with standard deviation in parentheses. The Tukey intervals for comparisons between storage conditions, acid concentrations, and techniques and among liners were 1.5, 1.5, 1.5, and 2.2 kg/cm², respectively.

+The Tukey interval for comparisons of Glasionomer Base Cement between conditioning times, acid concentrations, and techniques was 2.3 kg/cm².

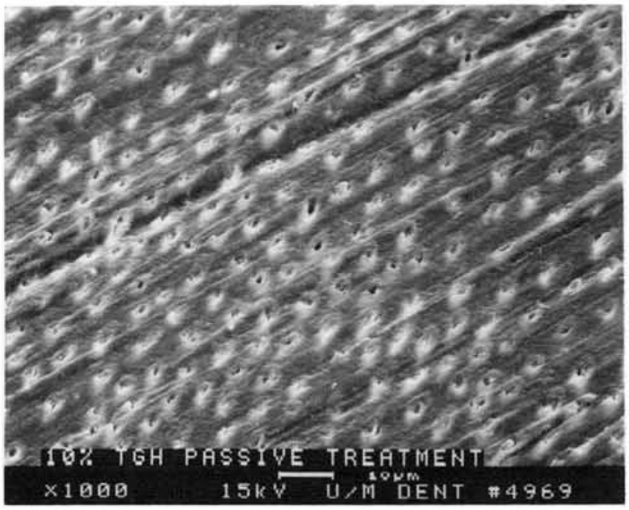

Fig. 1—Scanning electron photomicrograph of human dentin conditioned by 10% poly (acrylic acid) with passive conditioning for 30 seconds.

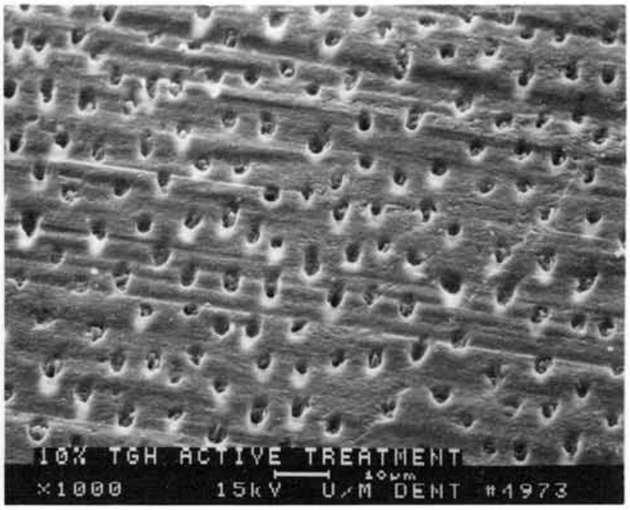

Fig. 2—Scanning electron photomicrograph of human dentin conditioned by 10% poly (acrylic acid) with active conditioning for 30 seconds.

Results.

Mean values and standard deviations of the bond strengths are reported in Table 2. Analysis of variance showed that significant differences existed among the means. Tukey intervals for comparisons among two conditioner concentrations, two 30-second conditioning techniques, two storage conditions, and three products calculated from the analysis of variance were 1.5, 1.5, 1.5, and 2.2 kg/cm², respectively, at the 95% level of confidence. The Tukey interval for comparisons among two conditioner concentrations, two conditioning techniques, and two etching times for Glasionomer Base Cement calculated from a second analysis of variance was 2.3 kg/cm² at the 95% level of confidence.

Bond strengths to untreated dentin after storage for 24 hours ranged from 19.0 to 21.7 kg/cm² for Glasionomer Base Cement, Cement/Liner, and Ketac-Bond, but dropped to a range of 4.9 to 9.7 kg/cm² after thermal cycling. Under conditions of 24-hour storage, conditioning produced no change or slight increase (10 to 20%) in bond strength for Cement/Liner and Ketac-Bond but 80 to 100% improvement in the bond strength for Glasionomer Base Cement.

Active conditioning with 10% acid resulted in bond strengths after 24-hour storage that ranged from 23.5 to 44.0 kg/cm², compared with values from 21.7 to 38.0 kg/cm² with active conditioning using 25% acid. In general, active conditioning resulted in statistically higher bond strengths than did passive conditioning.

Conditioning generally improved the resistance of the bonds to failure after thermal cycling compared with that of untreated dentin. Active conditioning with 10% acid resulted in bond strengths after thermal cycling that were in the range of 15.8 to 27.4 kg/cm² and were 80 to 320% higher than values for passive conditioning under these conditions.

Conditioning for 30 seconds produced 50 to 60% higher bond strengths for Glasionomer Base Cement than conditioning for 10 seconds, except for active conditioning for 30 seconds with 25% acid for which the bond strength was slightly lower.

Bond failures observed for the 24-hour storage period occurred adhesively at the liner-dentin interface for Ketac-Bond and Glasionomer Base Cement, but were mixed (adhesive and cohesive) for Cement/Liner. After thermal cycling, all bond failures of the liners occurred adhesively at the liner-dentin interface.

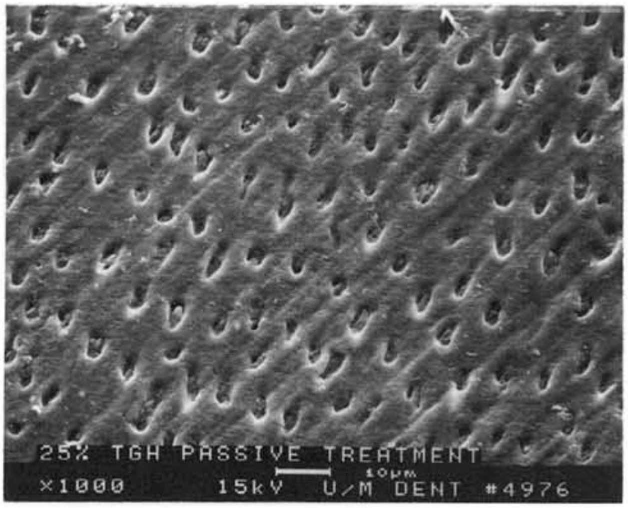

Fig. 3—Scanning electron photomicrograph of human dentin conditioned by 25% poly (acrylic acid) with passive conditioning for 30 seconds.

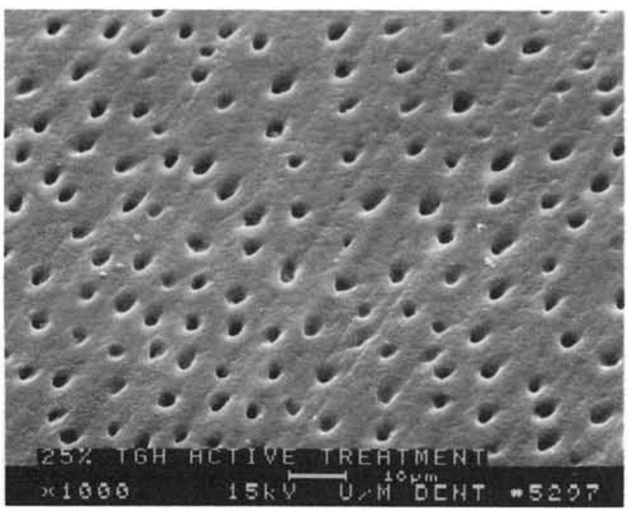

Fig. 4—Scanning electron photomicrograph of human dentin conditioned by 25% poly (acrylic acid) with active conditioning for 30 seconds.

Scanning electron photomicrographs of human dentin conditioned passively and actively for 30 seconds with concentrations of 10 and 25% poly (acrylic acid) are shown in Figs. 1 to 4, respectively. The dentin tubules were opened to a much lesser extent by passive conditioning with 10% acid and to a much greater extent by active conditioning with 25% acid.

Discussion.

Conditioning cut-dentin passively (without rubbing) with a low concentration (10%) of poly (acrylic acid) for a short time (10 seconds) is meant to remove the smear layer without increasing dentin permeability caused by removing the material blocking the dentinal tubules. While it is desirable to remove the smear layer, it is probably undesirable to over-condition or etch the dentin such that chemical groups (calcium or phosphate ions) that might be involved in the adhesion of the glass-ionomer cement are removed. Smith and Ruse (1987) have shown that etching bovine dentin can deplete surface calcium which might cause variable bonding.

Bond failures occurred at the liner-dentin interface except for Cement/Liner when stored for 24 hours. These data suggest that the cohesive strength of Cement/Liner may be less than that of Ketac-Bond and Glasionomer Base Cement. Compressive strengths at 24 hours of Glasionomer Base Cement, Ketac-Bond, and Cement/Liner were measured to be 1850, 1650, and 1090 kg/cm^2, respectively. Tensile strengths of glass-ionomer bases were not measured but have been reported to be 35 to 40 kg/cm^2 (Craig, 1988), the same order of magnitude as the bond strengths reported in this study.

Clinically, the bond strength data in this study suggest that active conditioning is preferable to passive conditioning with 10% poly (acrylic acid) for 30 seconds. Further testing is required to determine whether the increased bond strength is achieved at the expense of increased microleakage.

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