Parameters affecting *in vitro* bond strength of composites to enamel and dentin

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Abstract– *In vitro* tensile bond strengths of Photo Clearfil Bright (PB) and Clearfil Photo Posterior (CP) with Clearfil Photo Bond to human enamel and dentin were determined by a truncated cone bond test. Parameters tested were: four substrates (etched enamel; unetched dentin, D1; dentin conditioned with 10% polyacrylic acid, D2; and dentin etched with phosphoric acid, D3); two techniques (bonding agent prepolymerized, and bonding agent and composite polymerized together); and two storage times in 37°C water (one day and one week). Bond strengths of PB and CP to enamel ranged from 15 to 25 MN/m^2 and 19 to 24 MN/m^2, respectively. Bond strengths of PB to dentin ranged from 10.2 to 13.6 MN/m^2 for D1, 8.2 to 10.5 MN/m^2 for D2, and 5.5 to 7.8 MN/m^2 for D3. Bond strengths of CP to dentin ranged from 7.6 to 12.3 MN/m^2 for D1, 6.8 to 9.5 MN/m^2 for D2, and 4.6 to 6.7 MN/m^2 for D3. Bond strengths were higher to enamel than to dentin. The highest bond strengths to dentin were to unetched dentin, followed by conditioned dentin, and then etched dentin. Polymerization techniques and storage times did not affect the bond strengths.

Over 30 years have passed since Buonocore proposed acid-etching of enamel to achieve bonding between acrylic resin and enamel (Buonocore, 1955). The acid-etch technique, accompanied by a bonding agent, has become a routine procedure for composite resin restorations. Better adhesion between enamel and composites is expected to be effective in preventing marginal leakage, strengthening the tooth, and allowing for a more conservative cavity preparation.

Even with the acid-etch technique, early bonding agents did not exhibit bonding to dentin as they did to enamel. Generally, dentin had a lower bond strength with composites than did enamel. Recently, many commercial products have provided phosphonate dentin bonding agents that are claimed to adhere to dentin mechanically and chemically (Gwinnett, 1985; Council on Dental Materials, Instruments, and Equipment, 1987). Other newly developed bonding systems have also been reported (Bowen *et al.*, 1982; Bowen and Cobb, 1983; Munksgaard and Asmussen, 1984; Tagami *et al.*, 1987), some of which require complicated application procedures.

Although a number of bonding techniques have been studied, an agreed-upon method to achieve optimal bond strength between dentin and composites has not been established. An American Dental Association report (Council on Dental Materials, Instruments, and Equipment, 1987) stated that no consensus has been reached on the clinical effectiveness of existing dentin bonding agents.

This study investigated the effects of several parameters on the *in vitro* tensile bond strength of two light-cured composites with a light-cured bonding agent to human teeth, by use of a truncated cone bond test. The parameters were: four substrates (etched enamel, unetched dentin, dentin conditioned with 10% polyacrylic acid, and dentin etched with phosphoric acid), two polymerization techniques (bonding agent prepolymerized, and bonding agent and composite polymerized together), and two storage times in 37°C water (one day and one week).

MATERIALS AND METHODS

Experimental parameters of four substrates, two materials, two polymerization techniques, and two storage conditions are summarized in Table 1. Enamel samples were prepared from extracted human mandibular incisors embedded in

TABLE 1
SUMMARY OF EXPERIMENTAL CONDITIONS

Parameter	Code	Summary
Substrate	–	Enamel etched with phosphoric acid
	D1	Unetched dentin
	D2	Dentin conditioned with 10% polyacrylic acid
	D3	Dentin etched with phosphoric acid
Technique	–	Bonding agent prepolymerized
	–	Bonding agent and composite polymerized together
Material	PB	Photo Clearfil Bright
	CP	Clearfil Photo Posterior
Storage Time	–	1 day in 37°C water
	–	1 week in 37°C water

TABLE 2
BOND STRENGTHS OF COMPOSITE RESINS TO ENAMEL

Photo Clearfil Bright (PB)		
	1 day in water	1 week in water
Prepolymerized	20 (11)*	15 (8)
Polymerized Together	20 (7)	25 (7)
Clearfil Photo Posterior (CP)		
	1 day in water	1 week in water
Prepolymerized	22 (8)	22 (10)
Polymerized Together	24 (14)	19 (4)

*Mean of five replications in MN/m² with standard deviation in parentheses. The Tukey interval for comparisons among two composites, two polymerization techniques, and two storage times at the 95% level of confidence was 6 MN/m².

polyester resin. Dentin samples were prepared from human molars. The facial surface of each tooth was ground flat under water by means of 600-grit silicon carbide paper. All samples were stored in 37°C distilled water before use.

Enamel samples were etched with phosphoric acid (K-Etchant, batch EG012, Kuraray Co., Ltd., Osaka, Japan) for 60 sec, rinsed thoroughly, and dried with filtered compressed air. Dentin samples were prepared by three different methods as summarized in Table 1. Group D1 was not etched. Group D2 was conditioned with polyacrylic acid (Dentin Conditioner, batch 070561, G-C Dental Industrial Corp., Tokyo, Japan) for 20 sec. Group D3 was etched with phosphoric acid (K-Etchant) for 60 sec. After treatment, dentin samples were rinsed and dried like the enamel samples.

Adhesive tapes with 3- or 5-mm holes were placed on the enamel and dentin surfaces, respectively. A light-cured bonding agent (Clearfil Photo Bond; universal, 201; catalyst, 101; Kuraray Co., Ltd., Osaka, Japan) was applied to the surface, and the residual solvent was blown off with filtered compressed air. The bonding agent was polymerized with a Marathon (Den-Mat Corp., South El Monte, CA) curing unit for 30 sec by two methods: bonding agent prepolymerized before placement of composite, and bonding agent and composite polymerized together.

The two light-cured composites tested were Photo Clearfil Bright (PB, batch TM-HBS-111, Kuraray Co., Ltd., Osaka, Japan) and Clearfil Photo Posterior (CP, batch HPS-10U1, Kuraray Co., Ltd., Osaka, Japan). An inverted truncated cone die made of silicone rubber was placed over the hole in the tape and filled with composite resin. An incremental curing technique was used. Each increment was cured for 30 sec. Further irradiation for 60 sec was done after the mold was removed. Samples were stored in distilled water at 37°C for two time periods, one day and one week. Teeth were not reused. Each group had five replications.

Debonding of samples was accomplished with a testing machine (Instron Model TT-BM, Instron Corp., Canton, MA) at a cross-head speed of 0.05 cm/min, by the truncated cone bond test described by Barakat and Powers (1986). Bond strengths were calculated from the load at failure divided by the nominal area (enamel – 7.07 mm² and dentin – 19.6 mm²).

Mean values and standard deviations were calculated. Data were analyzed in three separate groups (enamel, dentin with PB, and dentin with CP) by analysis of variance (Dalby, 1968). Means were compared with Tukey's multiple comparison intervals (Guenther, 1964) at the 95% level of confidence.

RESULTS

Table 2 shows the tensile bond strengths of two composites to etched enamel. Bond strengths of PB ranged from 15 to 25 MN/m². When the bonding agent was prepolymerized, the value of 20 MN/m² for one-day storage decreased to 15 MN/m² for one-week storage. The values improved by polymerization of bonding agent and composite together at one-week storage, but not at one-day storage. Bond strengths of CP to enamel ranged from 19 to 24 MN/m² and were not affected by technique or storage time. The Tukey interval for comparisons among two composites, two polymerization techniques, and two storage times at the 95% level of confidence was 6 MN/m².

Tables 3 and 4 show the tensile bond strengths of PB and CP to dentin. Bond strengths of PB ranged from 10.2 to 13.6 MN/m² for unetched dentin, 8.2 to 10.5 MN/m² for dentin conditioned with 10% polyacrylic acid, and 5.5 to 7.8 MN/m² for dentin etched with phosphoric acid. While statistical analysis showed no significant differences between techniques and storage times, the bond strength to dentin was significantly affected by surface treatment. The Tukey intervals for comparisons among three surface treatments, two polymerization techniques, and two storage times at the 95% level of confidence were 2.7, 1.9, and 1.9 MN/m², respectively.

Bond strengths of CP varied from 7.6 to 12.3 MN/m² for unetched dentin, 6.8 to 9.5 MN/m² for conditioned dentin, and 4.6 to 6.7 MN/m² for etched dentin. Significant differences were not observed between techniques and storage times. Only

TABLE 3
BOND STRENGTHS OF PHOTO CLEARFIL BRIGHT (PB) TO DENTIN

Storage for 1 day in water at 37°C			
	Unetched (D1)	Conditioned (D2)	Etched (D3)
Prepolymerized	10.2 (3.0)*	10.4 (3.4)	5.9 (1.7)
Polymerized Together	13.5 (3.4)	10.5 (1.4)	7.8 (4.1)
Storage for 1 week in water at 37°C			
	Unetched (D1)	Conditioned (D2)	Etched (D3)
Prepolymerized	12.7 (3.7)	8.2 (3.2)	5.5 (3.1)
Polymerized Together	13.6 (7.8)	9.5 (3.7)	5.9 (2.6)

*Mean of five replications in MN/m² with standard deviation in parentheses. The Tukey intervals for comparisons among three surface treatments, two polymerization techniques, and two storage times at the 95% level of confidence were 2.7, 1.9, and 1.9 MN/m², respectively.

surface treatment affected the bond strengths of CP to dentin. A significant difference was observed between bond strengths to unetched dentin and dentin etched with phosphoric acid. The Tukey intervals for comparison among three surface treatments, two polymerization techniques, and two storage times at the 95% level of confidence were 2.5, 1.7, and 1.7 MN/m^2, respectively.

TABLE 4
BOND STRENGTHS OF CLEARFIL PHOTO POSTERIOR (CP) TO DENTIN

Storage for 1 day in water at 37°C	Unetched (D1)	Conditioned (D2)	Etched (D3)
Prepolymerized	8.9 (1.9)*	8.2 (3.6)	6.4 (2.2)
Polymerized Together	7.6 (2.4)	6.8 (4.1)	5.5 (2.2)
Storage for 1 week in water at 37°C	**Unetched (D1)**	**Conditioned (D2)**	**Etched (D3)**
Prepolymerized	12.3 (5.3)	9.5 (3.8)	6.7 (3.1)
Polymerized Together	10.9 (4.6)	7.4 (2.8)	4.6 (1.6)

*Mean of five replications in MN/m^2 with standard deviation in parentheses. The Tukey intervals for comparison among three surface treatments, two polymerization techniques, and two storage times at the 95% level of confidence were 2.5, 1.7, and 1.7 MN/m^2, respectively.

DISCUSSION

According to the results of this study, polymerization techniques and storage times did not affect the bond strengths of composite to either enamel or dentin. Another *in vitro* study (Mowery *et al.*, 1986) employing simultaneous and independent polymerization techniques also found no significant difference between the enamel shear bond strengths of the two techniques. A thermal stress test was not conducted in this study.

The average bond strengths to etched enamel of both composites were higher than to dentin with any surface treatment, although statistical comparisons between bond strengths to enamel and dentin were not made because of differences in the variances. Except for two testing conditions, the average bond strengths of both composites to etched enamel were approximately 20 MN/m^2, which are considered to be strong enough against debonding stress caused by polymerization shrinkage and able to prevent marginal opening (Komatsu and Finger, 1986).

The effects of the three treatments (D1 to D3) to dentin on tensile bond strength were analyzed. With PB, average bond strengths decreased as much as 33% for dentin conditioned with polyacrylic acid (D2), and 50% for dentin etched with phosphoric acid (D3) when compared with unetched dentin (D1). With CP average bond strengths decreased as much as 20% for dentin conditioned with polyacrylic acid (D2), and 42% for dentin etched with phosphoric acid (D3) when compared with unetched dentin (D1). Both composites had the highest bond strength to unetched dentin among the three testing conditions, followed by conditioned dentin and then etched dentin. The large standard deviation might conceal the difference between unetched and conditioned dentin with CP.

It has been reported that bond strength to dentin was not improved by etching with phosphoric or citric acids (Retief *et al.*, 1986a; Torney, 1978; Solomon and Beech, 1985; Odén and Øilo, 1986). Other studies have shown that various chemical agents were effective for demineralization and cleaning of dentin surfaces covered by a smear layer (Silverstone, 1975; Brännström and Johnson, 1974; Duke *et al.*, 1985; Retief *et al.*, 1986b; Meryon *et al.*, 1987).

A bonding agent can easily penetrate dentin tubules and form resin tags, which result in mechanical interlocking. Clearfil Photo Bond contains 2-methacryloxyethyl phenyl phosphoric acid, which is proposed to bond chemically to dentin (Nakabayashi, 1985). The proposed bonding mechanism is that phosphate can bond to calcium in the hydroxyapatite of the dentin. A surface treatment that reduces the mineral fraction may affect the bond strength of this bonding agent to dentin.

According to the quantitative study by Meryon *et al.* (1987), the effect on the smeared layer and dentinal tubules was higher with 37% phosphoric acid than with 25% polyacrylic acid. Phosphoric acid may be stronger than needed. Direct application of strong chemical agents may cause dissolution of the mineral components of dentin. A weakened substrate may show inferior bonding properties.

Clearly, etching of enamel is necessary for an optimal composite restoration. Many attempts have been made to achieve better adhesion to dentin. Because the smear layer contains debris, micro-organisms, and other contaminants, it cannot be left untreated. Thus, the improved bonding achieved in this study with unetched dentin may not have direct clinical application.

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