In vitro bond strength of cements to treated teeth

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Abstract

Adhesive bond strengths of glass ionomer and polycarboxylate cements to human enamel and dentine were measured *in vitro* on untreated surfaces and on surfaces treated with cement liquid or polyacrylic acid, ferric oxalate, and ferric chloride. The bond strengths of two cements to enamel and dentine were increased by as much as 183 per cent by the use of surface treatments of polyacrylic acid and ferric oxalate.

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Introduction

The bonding of the cement to enamel and dentine is one of the factors that determines the success of a cemented restoration. Several investigators (Buonocore,[1] Beech,[2] Hotz et al.,[3] Reteif,[4] and Sheykholeslam et al.[5]) have used acid etching to increase the bond strength to enamel, but this technique has not been successful for dentine. Powis et al.[6] showed that ferric chloride (2 per cent) increased the bond strength of glass ionomer cement to enamel by 40 per cent and to dentine by 70 per cent. Polyacrylic acid increased bond strength to enamel and dentine by about 120 per cent. Bowen,[7] Jedrychowski et al.,[8] Peddy, [9] and Shalabi et al.[10] used ferric chloride as a surface treatment in conjunction with different polycarboxylate and glass ionomer cements. The results varied from no improvement to 200 per cent improvement in bond strength. Bowen et al.[11,12] used ferric oxalate (5 per cent) as a surface treatment to obtain a 50 per cent increase in bond strength of a composite to enamel and a 700 per cent increase with dentine.

The purpose of this study was to investigate bond strengths of several cements to enamel and dentine *in vitro* using untreated tooth structure and tooth structure treated with cement liquid or polyacrylic acid, ferric chloride, and ferric oxalate.

Materials and methods

Codes, names, types, batch numbers, and manufacturers of the cements tested are listed in Table 1. The cements were mixed according to the manufacturer's instructions.

Freshly extracted third molars were stored in distilled water at room temperature until use. Each tooth was invested in a block of cold-cured resin with the buccal or lingual surface exposed. Flat surfaces of the teeth were prepared and polished with wet carborundum paper (240 and then 600 grit) and stored in water.

The tooth surfaces (enamel and dentine) were prepared according to the following conditions: (1) untreated, (2) etched with cement liquid, (3) treated with 15 per cent ferric chloride,‡ and (4) treated with 5 per cent ferric oxalate.§ For the two anhydrous cements (A and C), a commercial polyacrylic acid‖ was used as the cement liquid (condition 2). All surface treatments were applied to enamel and dentine for 60 seconds. The surfaces were then washed for 10 seconds and blown dry with air for 10 seconds before the cements were applied. Five samples were prepared for each cement and condition. Scanning electron micrographs were made of the treated surfaces of enamel and dentine.

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‖**Durelon Cement Liquid,** LA14210. Espe-Premier, Norristown, PA, USA.

Table 1. Codes, product names, batch numbers, powder/liquid ratios, and manufacturers of the cements tested

Code	Product	Type	Batch numbers	P/L ratio	Manufacturer
A	Tylok-Plus*	Anhydrous polycarboxylate	Powder 022483	5.0/1.0	L.D. Caulk Co. Milford, DE, USA.
B	Liv Cenera	Polycarboxylate	Powder 190431 Liquid 190431	2.0/1.0	G-C Dental Industrial Corp. Tokyo, Japan.
C	Biocem†	Anhydrous glass ionomer	Powder 020983	3.3/1.0	L.D. Caulk Co.
D	Lining cement	Glass ionomer	Powder 090731 Liquid 060731	1.2/1.0	G-C Dental Industrial Corp.

*Distributed as Poly F Plus.
†Distributed as Aquacem.

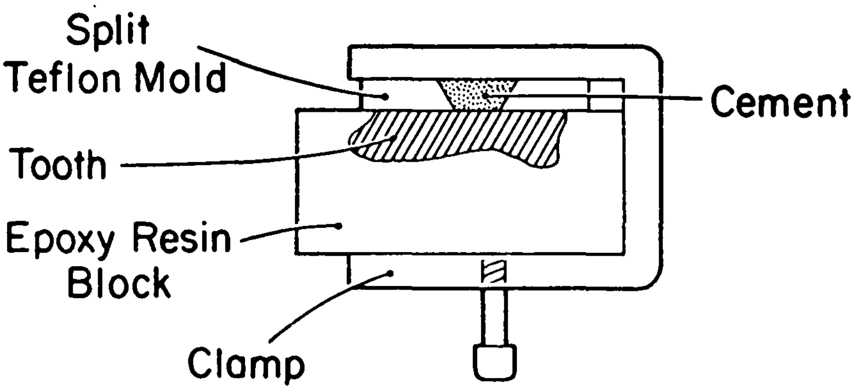

Fig. 1.–Assembly used to prepare bond strength specimens.

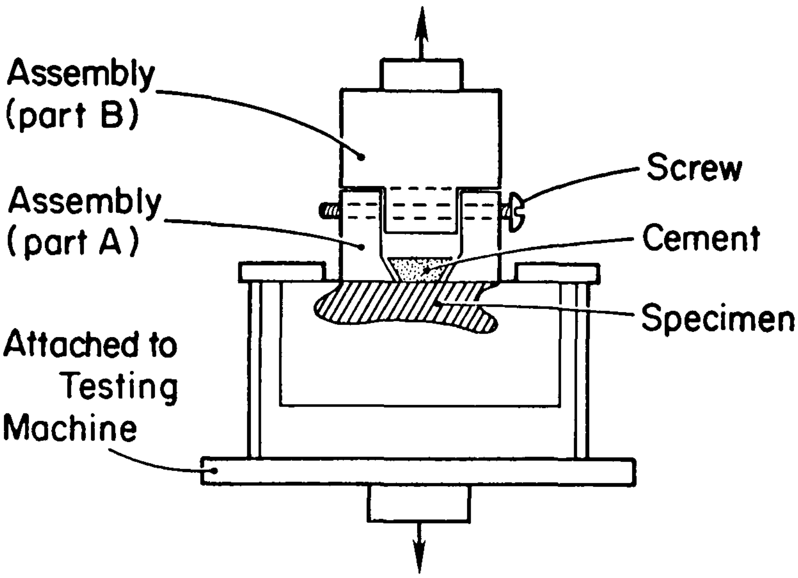

Fig. 2.–Assembly used to test bond strength specimens in tension.

Table 2. Tensile bond strengths (MPa) of cements bonded to treated enamel and dentine

Code	Substrate	Treatment			
		Untreated	Cement† liquid	Ferric chloride	Ferric oxalate
A	Enamel	3.7 (1.0)*	7.3 (0.7)	4.2 (2.2)	4.5 (0.7)
	Dentine	2.0 (0.8)	5.7 (1.9)	2.1 (1.4)	4.1 (1.1)
B	Enamel	3.7 (1.0)	3.4 (1.1)	3.4 (0.6)	2.9 (0.5)
	Dentine	1.3 (0.5)	1.7 (0.4)	1.9 (1.0)	2.3 (0.5)
C	Enamel	3.2 (1.3)	4.6 (1.0)	4.5 (0.4)	5.2 (1.3)
	Dentine	4.1 (0.6)	2.9 (0.7)	3.1 (0.8)	5.4 (2.0)
D	Enamel	4.1 (1.2)	3.9 (0.5)	3.2 (0.9)	2.7 (0.5)
	Dentine	2.0 (0.7)	1.9 (0.6)	2.3 (0.7)	2.1 (0.9)

*Mean values with standard deviations in parentheses. The Tukey interval for comparisons of means was 1.2 MPa among cements and treatments and was 0.6 MPa between substrates at the 95 per cent level of confidence.
†A commercial polyacrylic acid was used for the anhydrous materials, A and C.

The assembly used to prepare the bond strength specimens is illustrated in Fig. 1. After the cement had set for 15 minutes, the bonded tooth was removed from the assembly and stored in distilled water at 37° for 24 hours. Excess cement was removed from the bonding area before testing. The specimens were then subjected to a tensile load perpendicular to the tooth surface using the loading device illustrated in Fig. 2. The crosshead of the testing machine¶ moved at a speed of 0.1 mm per minute. The tensile bond strength was calculated as the failure load divided by the nominal area of the cement.

Means and standard deviations were calculated for each cement and treatment condition. The data were analysed by analysis of variance[13] using a factorial design. Means were compared by a Tukey interval[14] calculated at the 95 per cent level of confidence. Differences between two means that were larger than the Tukey interval were statistically significant.

Results

Means and standard deviations of tensile bond strengths are listed in Table 2. Effects of treatment and substrate on bond strengths of cements A through D were studied by analysis of variance. The coefficient of variation for these data was 30 per cent. Values of Tukey intervals also are reported in Table 2. Scanning electron micrographs of representative treated surfaces of enamel and dentine are shown in Figures 3 to 5.

Bond strengths of cements A to D to untreated teeth ranged from 1.3 to 4.1 MPa for dentine and from 3.2 to 4.1 MPa for enamel. Bond strengths to untreated enamel were greater than those to untreated dentine for cements A, B, and D. For cement C, the bond strength to dentine was greater.

Bond strengths were improved by the treatment of the enamel and dentine for cements A and C. For the anhydrous polycarboxylate cement (A), the use of a polyacrylic acid as a surface treatment resulted in 96 and 183 per cent increases in the bonding to enamel and dentine, respectively. Treatments with ferric chloride and ferric oxalate increased the bond strength but the increase was only significant for the bond strength of A to dentine modified with ferric oxalate.

For the anhydrous glass ionomer cement (C), the most effective surface treatment was ferric oxalate. Increases of 65 and 32 per cent were observed in the bond strengths to enamel and dentine. Treatments with polyacrylic acid and ferric chloride improved bonding to enamel but decreased bonding to dentine compared to untreated surfaces.

The surface treatments did not increase the bond strengths of cements B and D to enamel or dentine significantly.

Discussion

The bond strength specimens were designed in the shape of an inverted, truncated cone to increase the probability of bond failure at the cement-tooth interface and to minimize failure caused by gripping the specimen. All bond failures occurred at the cement-tooth interface. The elimination of failures caused by gripping the specimen makes the truncated cone bond test useful when factors such as an opposing substrate (a casting) and film thickness of the cement are not of interest.

Bond strengths to untreated enamel by the polycarboxylate and glass ionomer cements (A-D)

¶Model TT-BM. Instron Corporation, Canton, MA, USA.

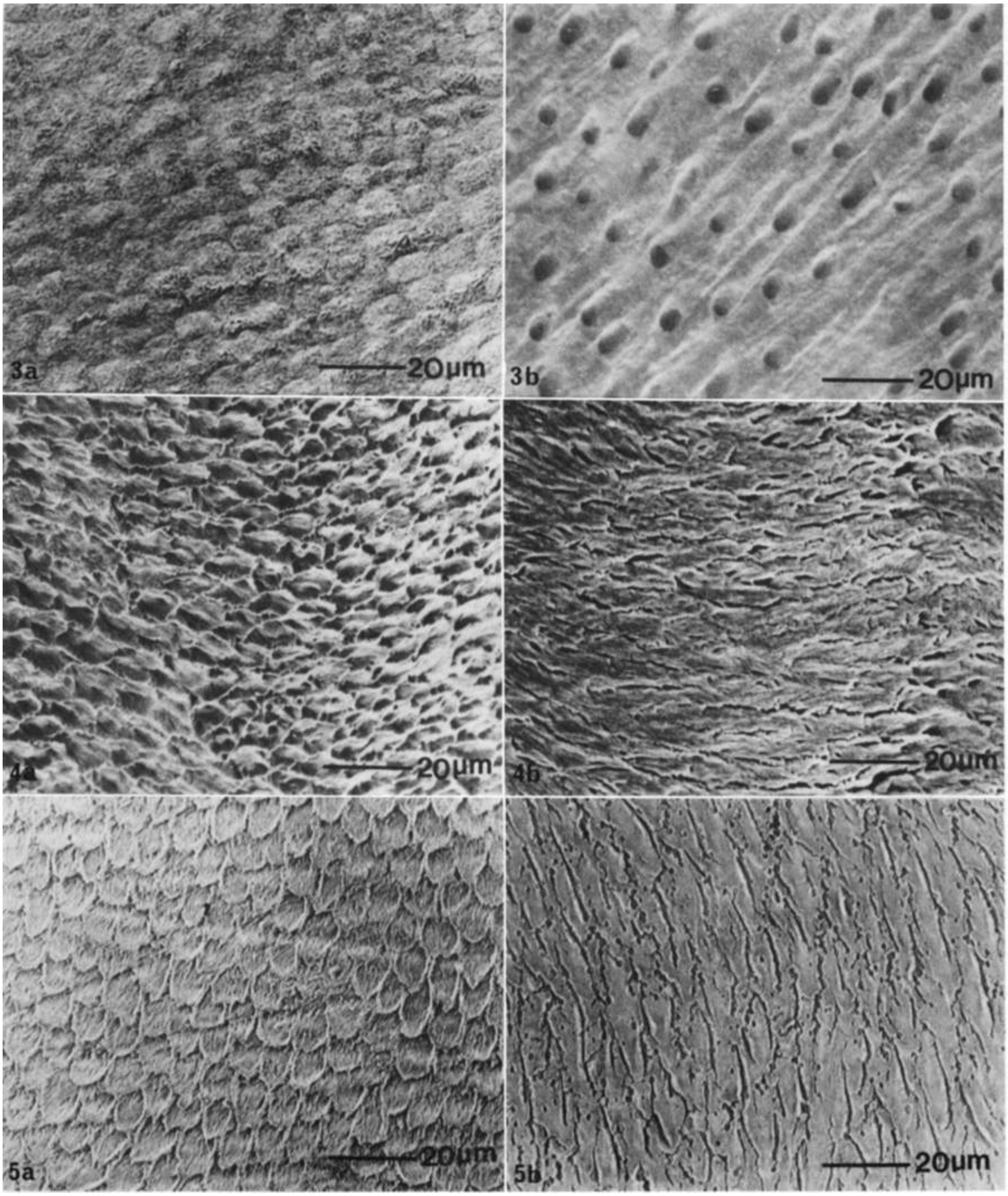

Fig. 3.—Scanning electron micrograph of enamel (a) and dentine (b) treated with a commercial polyacrylic acid.

Fig. 4.—Scanning electron micrograph of enamel (a) and dentine (b) treated with 15 per cent ferric chloride.

Fig. 5.—Scanning electron micrograph of enamel (a) and dentine (b) treated with 5 per cent ferric oxalate.

were similar to each other and to values reported by others.[6-12]

Bond strengths to untreated dentine by cements A to D were lower than values for enamel except for cement C which had a higher bond strength to dentine.

The treatments of polycarboxylic acid, ferric chloride, and ferric oxalate resulted in some improvements in bond strength for both enamel and dentine. Treatment by polyacrylic acid supplied with a commercial polycarboxylate cement improved the bond strength of the anhydrous cements A and C, whereas treatments with the polycarboxylic acid supplied with cements B and D were not effective.

Observation of the treated surfaces by scanning electron microscopy indicated differences in the etching patterns of enamel and dentine by the treatments. The polyacrylic acid etched the enamel and dentine mildly as shown in Fig. 3. The ferric chloride etched both enamel and intratubular dentine strongly forming a fissured surface (Fig. 4). Enamel etched by ferric oxalate (Fig. 5) was intermediate to that etched by polyacrylic acid and ferric chloride. The etched dentine was similar to that etched by ferric chloride. Both ferric chloride and ferric oxalate appeared to etch preferentially the organic phase of dentine. Inconsistent improvement in bond strength associated with the 15 per cent solution of ferric chloride used in this study may be the result of the severe etch produced. A milder etch may produce an improved bond as reported by Powis et al.[6]

The data suggest there is no universal surface treatment among those tested. For some cements, certain treatments can significantly improve bond strength to enamel and dentine. The mechanisms by which bond strength is improved were not studied in this experiment. Biological implications of etching dentine also were not considered in this study.

Conclusions

Adhesive bond strengths of glass ionomer and polycarboxylate cements to human enamel and dentine were measured *in vitro* on untreated surfaces and on surfaces treated with cement liquid or polyacrylic acid, ferric oxalate, and ferric chloride. The bond strengths of two cements to enamel and dentine were increased by as much as 183 per cent by the use of surface treatments of polyacrylic acid and ferric oxalate.

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