



Effects of Different Tooth-Conditioning Agents on Bond Strength of A Resin-Modified Glass-Ionomer Cement to Enamel

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ABSTRACT

The purpose of this study was directed to evaluate the effects of different tooth conditioning agents on shear bond strength of resin-modified glass– ionomer cement (Fuji III LC) to enamel as well as on dissolution of calcium ions from the enamel surfaces. **Methods.** The enamel surfaces of lower incisors were treated with 10 and 20% polyacrylic acid, 12% citric acid and 35% phosphoric acid for 20s. Fuji III LC was applied to the etched enamel surfaces, and the shear bond strength of each specimen was measured using an Instron Universal Testing Instrument. The amounts of calcium ions dissolved from the treated enamel surfaces were also measured using a polarized Zeeman atomic absorptiometer. **Results.** In specimens pretreated with distilled water, 10% polyacrylic acid, 20% polyacrylic acid, 12% citric acid and 35% phosphoric acid, the mean values of shear bond strength were 5.5, 12.5, 15.2, 15.2 and 15.1 MPa, respectively, and the amounts of Ca² dissolved from the enamel surfaces were 5.6, 41.4, 88.5, 131.6 and 588.3 mg/cm², respectively. **Conclusions.** The adhesion of a resin-modified glass–ionomer cement to enamel was significantly improved by the use of tooth-conditioning agents. Especially, treatments of an enamel surface with 20% polyacrylic acid results in good shear both strength and relatively small degree of enamel erosion.

INTRODUCTION

Sealing of pits and fissures has generally been accepted to have significant effects on the prevention of occlusal caries.¹⁻⁴ Resin-based and glass– ionomer cements are currently used widely in clinical practice. Glass–ionomer cement is known to have the characteristic of adhering chemically to the tooth structure,⁵⁻⁷ and there have been many reports on the retention of glass –ionomer cement applied to pits and fissures in clinical practice. High retention rates of glass –ionomer cement have been demonstrated in clinical trials.^{3,8,9} McLean and Wilson³ reported 84% complete clinical retention after 1 year and 79% after 2 years.

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McKenna and Grundy⁸ showed a retention rate of 82.5% after 1 year. Mills and Ball⁹ also reported a similar retention rate of 81% after 1 year and 83% after 2 years. Other investigators,^{10 - 14} however, have pointed out that a glass-ionomer cement used to cover pits and cements may not have full retention during the first 6 months to 1 year after application. Shimokobe et al.¹⁰ reported that glass-ionomer cement completely disappeared within 6 months. Boksman et al.¹¹ reported that cement was absent in 94% of cases after 6 months. Mejía and Mjor¹² reported that total loss of cement occurred in 61% of cases after 6–12 months and in 84% of cases after 30–36 months. Forss et al.¹³ and Williams et al.¹⁴ also reported very low retention rates in their 2–4 year clinical trials. Accordingly, it was suggested that glass-ionomer cement requires reapplication or replacement to maintain a complete seal of the occlusal surface.¹⁵ In all of these studies, however, the caries-preventive effect of the cement was significant. Treatment of enamel surfaces with an acid prior to cement application has not been included in the conventional procedure for the use of glass-ionomer cement. This may have been a reason for the low retention rates of glass-ionomer cements. However, there have been no reports on appropriate pretreatment of enamel surfaces when sealing pits and fissures with glass-ionomer cement.

The purpose of this study was to evaluate the effects of tooth-conditioning agents on bond strength of resin-modified glass-ionomer cement to enamel as well as on dissolution of calcium ions from the enamel surfaces.

MATERIALS AND METHODS

A total number of 55 sound non carious human extracted lower incisors, free of cracks and any developmental defects, were used in this study. The teeth washed under running tap water to remove blood and debris, scaled to remove calculus and remnants of periodontal tissues, polished with fine pumice and soft rubber cups rotating at low speed under coolant. The teeth were stored in distilled

water at room temperature (37 C⁰) until use. The distilled water was changed daily. The roots of the teeth were removed and the center of the labial surface of each crown was cut into a 10 mm² section. The teeth were embedded in auto polymerizing polyester resin in a casting ring of 22 mm in diameter and 30 mm in height with the surface of the labial enamel parallel to the base of the molded polyester. The enamel surfaces were ground with a grinding paper disc of 180 grit (CARBIMETw; BUEHLER, Lake Bluff, IL, USA) and polished with polishing paper of 600 grit (CARBIMETw) using MINIMETw1000 (BUEHLER). The speed/force in grinding or polishing was 30 rpm/13.3 N for 5 min. The specimens were then stored in distilled water at 4 8C. The grounded enamel surfaces were washed with distilled water using an ultrasonic apparatus (ULTRASONICCLEANERw; SHARP,Osaka, Japan) prior to each experiment.

Table 1: Conditioning agents for pretreatment of enamel.

Conditioning Agents	Company Chief	Component
Dentin conditioner GC Company,	Tokyo 113 0033, Japan	10% polyacrylic Acid
Cavity	conditioner GC Company, Tokyo 113 0033, Japan	20% polyacrylic Acid
BONDWELL LC Conditioner	GC Company, Tokyo 113 0033, Japan	12% citric Acid
Scotchbond etchant	3M Dental Products, St Paul, MN 55144, USA	35% phosphoric Acid

Shear testing

Preparation of the experimental surfaces of the enamel consisted of 20 s etching with the conditioning agents listed in Table 1 followed by 10 s rinsing and air-drying. A teflon tube of 4.0 mm in diameter and 1.5 mm in height was placed on the etched enamel surface and gently filled with resin modified glass –ionomer cement (Fuji III LCw; GC Corporation, Tokyo, Japan) that had been mixed according to the instructions of the manufacturer and then subjected to light illumination (New Light VL-IIw; GC Corporation, Tokyo, Japan) for 30 s from the top of the teflon tube over a celluloid strip. The tip of the light curing unit was placed as close to the strip as possible. The intensity of the light source was 837 mW/cm² at the peak of wavelength.¹⁶ The teflon tube and the celluloid strip were removed after light-curing had been completed, and the rest was stored in distilled water at 37.0 8C for 24 h.^{17,18}

Each group consisted of eight samples. Thermal fatigue tests were then carried out using 500 cycles of thermal stress and the samples were kept alternately for 30 s in a water bath of either 5 or 55 8C in each trial.^{17,18} After the loading of thermal stress, Shear bond strength was measured using Universal Testing Machine (LR 5K LLOYD instruments, Ltd, Hampshire, UK) as shown in Figure(1).

The use of jig enabled a load to be applied parallel to the enamel surface at the cement –enamel interface. The blade was pulled up at a crosshead speed of 1 mm/min (Fig. 1). All samples were tested in this way to measure their shear bond strengths.^{17,18} Fractured surfaces of the samples were coated with gold, and the type of fracture was examined using an IBAS image analysis system. The percentage of remaining cement on the experimental tooth surface was estimated. The type of fracture was assessed with a modified adhesive remnant index (ARI).^{19–21} The ARI scale has a range of 5 to 1:5 in the case of no cement remaining on the enamel,⁴ in the case of less than 10% of cement remaining, 3 in the case of more than 10% but less than 90% of cement re-

maining,² in the case of more than 90% of cement remaining, and 1 in the case of all of the cement remaining on the enamel surface.

The ARI scores were also used as a more complex means of defining the site of bond failure between the enamel and the cement. Demineralized calcium ions (Ca²⁺) measurement for demineralized Ca²⁺ measurement, adhesive tape with a hole of 6.0mm in diameter was attached to the enamel surface to regulate the area for conditioning. The enamel surfaces were then etched for 20 s using 10 ml of each conditioning agent (Table 1). The etching process was stopped by soaking the whole sample in 20 ml of 1% weight lantern chloride solution.

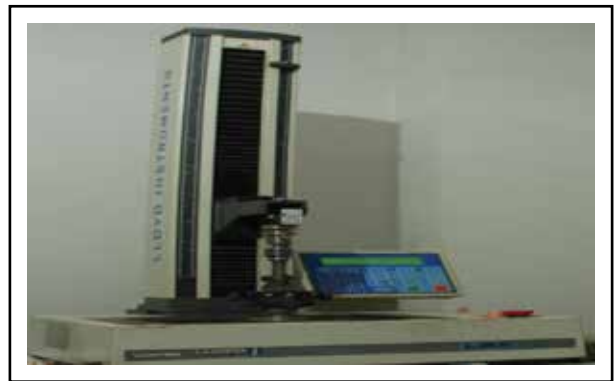


Fig. (1) Instron Universal Testing Machine (LR 5K LLOYD instruments, Ltd, Hampshire, UK).

The solution was immediately shaken for 20 s using an ultrasonic apparatus (SONIFIER Model 185w; Branson Sonic Power, Danbury, CT, USA) and supplemented with 2 ml of hydrochloric acid at the final concentration of 0.1N to prevent precipitation of calcium. The 4rCa²⁺ concentration in the solution was measured using a polarized Zeeman atomic absorptiometer (Z-8100; HITACHI, Tokyo, Japan), and the original amount of demineralized Ca²⁺ dissolved from the enamel surfaces (mg/cm²) was calculated. Each group consisted of three samples. Statistical analysis, The experimental data were analyzed statistically using one-way ANOVA and Fisher's PLSD.

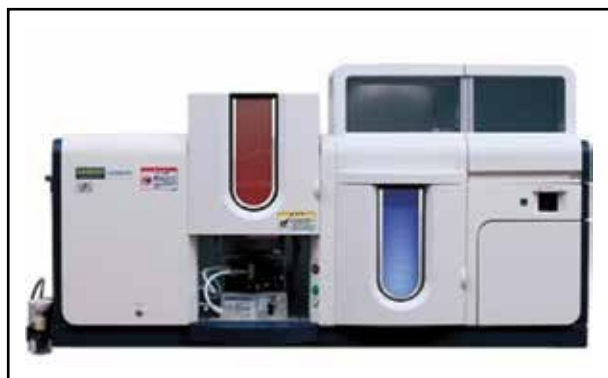


Fig. (2) Polarized Zeeman Atomic Absorption Spectrophotometer ZA3000 Series (Asuit university, Faculty of Sciences).

RESULTS

Shear bond strength

Table 2 shows the shear bond strength of each group. Pretreatment with any of the conditioning agents used in this study resulted in an increase in shear bond strength compared to that without pretreatment ($p < 0.01$). The shear bond strength of the group pretreated with Dentin Conditioner was significantly lower than those of the other groups ($p < 0.01$). No statistically significant differences were found among the other three groups (Cavity Conditioner w, BONDWELL LC Conditionerw and Scotchbond Etchantw). Adhesive residual index (ARI) The ARI scores for the five groups tested are presented in. There were no significant differences between the five groups, although there were some different distributions of ARI scores in five groups. Demineralized calcium ions (Ca21) measurement shows the results of demineralized Ca2 measurement. The amount of demineralized Ca2 in the group pretreated with deionized water was 5.6 mg/cm². This may have been Ca2 that contaminated the sample solution during experimental procedures such as storing and washing. This value, however, was significantly less than the values in other groups and considered to be the baseline value for the experiment. The amounts (means standard deviations) of demineralized Ca2

enamel surfaces exposed to Dentin Conditioner, Cavity Conditionerw, BONDWELL LC Conditioner and Scotchbond Etchant were 41.4, 3.8, 88.5, 3.1, 131.6, 10.7 and 588.3, 28.3 mg/cm², respectively. Statistically significant differences were found among these four groups ($p < 0.05$).

Table (2) Shear bond strength of each group

Conditioning agents	Shear bond strength (mpa)
Distilled water	5.5+ ₋ 1.3
Dentin conditioner	12.5 + ₋ 1.8
Cavity conditioner	15.2 + ₋ 1.4
Bondwell LC conditioner	15.2 + ₋ 1.0
Scotchbond Etchant	15.1 + ₋ 1.0

DISCUSSION

Glass-ionomer cement has the characteristic of adhering chemically to the tooth structure.⁵⁻⁷

It was, therefore, thought that preparation of an enamel surface prior to the cement application, such as enamel etching with phosphoric acid that is performed before the application of a resin-based cement, is not necessary for a glass-ionomer cement. However, some studies have shown that bond strength to enamel (or dentin) of a glass-ionomer cement used for filling was improved by tooth conditioning with an acid.²²⁻²⁴ Accordingly, it is possible that bond strength to enamel of a glass-ionomer cement used as a cement is also improved by surface treatment. The purpose of this study was to evaluate the effects of tooth-conditioning agents on bond strength of Fuji III LCw to enamel as well as on dissolution of calcium ions from the enamel surface. This study demonstrated that the shear bond strength of a resin-modified glass-ionomer cement to enamel increased significantly by using an acid, especially by using 20% polyacrylic acid, 12% citric acid or 35% phosphoric acid. The mean value of shear bond strength was obtained by pretreatment

of the enamel surfaces with 20% polyacrylic acid or 12% citric acid (15.2 MPa), while the mean value in the untreated group was only 5.5 MPa. The increase in shear bond strength by surface treatment coincides with the results of other studies,^{25–34} though it is difficult to compare our data directly with those of other studies because of differences in the experimental designs, such as thermal fatigue testing, regulation of the size of adhesion area and the cement material used. Shear testing of Fuji III LCw, which was applied to enamel after pretreatment with conditioning agents, frequently revealed cohesive/interfacial fracture, as indicated by the ARI scores, most of which were distributed 4 or 3. Studies by Gordan et al.³³ (using Fuji Bond LCw with 20% polyacrylic acid) and Dewji et al.³⁴ (using Fuji III LCw with 20% polyacrylic acid) also showed that both cohesive and interfacial fractures occurred at the interface between enamel and resin-modified glass-ionomer cement.

We also examined the dissolution of calcium ions from the enamel surfaces. The results showed that the amounts of calcium ions dissolved from enamel surfaces treated with distilled water, 10% polyacrylic acid, 20% polyacrylic acid, 12% citric acid and 35% phosphoric acid were significantly different ($p < 0.05$). The mean value of 5.6 mg/cm² in specimens pretreated with distilled water was considered to be a baseline concentration of calcium ions, possibly due to contamination during the procedure of ultrasonic stirring of the specimens.

The amount of Ca²⁺ dissolution in specimens treated with 20% polyacrylic acid was approximately two-times greater than that in specimens treated with 10% polyacrylic acid. The amount of Ca²⁺ dissolution in specimens treated with 12% citric acid was significantly greater than the amounts in specimens treated with the abovementioned two agents, and the greatest amount of Ca²⁺ dissolution was in specimens treated with 35% phosphoric acid.

The roughness of the enamel surface is thought to be related to micro-mechanical bond

strength,³⁵ and it is thought that the most effective conditioning agents for adhesion, such as citric acid and phosphoric acid, dramatically alter enamel surfaces. The results of Ca²⁺ dissolution indicate the degree of enamel erosion and surface roughness. However, no significant differences were found between shear bond strengths of specimens treated with 20% polyacrylic acid, 12% citric acid and 35% phosphoric acid. These findings suggest that adhesion of Fuji III LCw to enamel depends mainly on the chemical polar and ionic bond rather than on the micromechanical bond. Powis et al.²³ reported that the most effective surface conditioners to improve the adhesion of a glass-ionomer cement were high-molecular-weight substances containing a multiplicity of functional groups capable of hydrogen bonding, such as polyacrylic acid, and that these substances did not greatly disrupt the enamel surfaces, ensuring effective cleaning and wetting of the surfaces. They also reported that low-molecular-weight chelating agents, such as citric acid, that dissolve calciferous material and dramatically alter the surfaces were less effective. The retention rate of glass-ionomer cement is generally thought not to be high. Most studies,^{3,8,10,12–15} however, have shown that dental caries rarely occurred in pits and fissures sealed with glass-ionomer cement regardless of the retention rate. It is thought that the caries-preventive effect of glass-ionomer cement depends not only on retention within pits and fissures but also on fluoride release to adjacent tooth structures and the oral environment.^{13,36–38} Our previous study on the retention rates of resin-modified glass-ionomer cement (Fuji III LCw)³⁹ showed full retention rates of 87.5% by clinical examination and 50% by SEM examination at 12 months and 70.3% by clinical and 9.7% by SEM at 24 months. Secondary caries was detected in only one case at 18-month recall. In that clinical trial, 10% polyacrylic acid (Dentin Conditioner w; GC, Tokyo) was used as an enamel surface-conditioning agent.

The present study, which was designed to establish a clinical procedure that would improve

the retention rate, revealed that the use of some conditioning agents significantly increased the shear bond strength of resin-modified glass – ionomer cement to enamel. There were no differences in shear bond strengths of specimens treated with 20% polyacrylic acid (15.2 MPa), 12% citric acid (15.2 MPa) and 35% phosphoric acid (15.1 MPa). On the other hand, the degree of Ca² dissolution, i.e. the severity of enamel erosion, varied according to the conditioning agent used.

CONCLUSIONS

The purpose of this study was to evaluate the effects of tooth-conditioning agents on bond strength of resin-modified glass –ionomer cement to enamel as well as on dissolution of calcium ions from the enamel surfaces. In conclusion, polyacrylic acid is a suitable tooth conditioning agent for a resin-modified glass –ionomer cement. Treatment of an enamel surface with 20% polyacrylic acid results in good shear bond strength and relatively small degree of enamel erosion.

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