

Effect of Different Conditioning Protocols on the Adhesion of a Glass Ionomer Cement to Dentin

Hila Hajizadeh, DDS, MS; Marjaneh Ghavamnasiri, DDS, MS;
Mohammad Sadegh Namazikhah, DMD, MSED;
Sara Majidinia, DDS; Mahshid Bagheri, DDS



Abstract

Aim: The purpose of this study was to assess the effect of different surface treatments on the shear bond strength (SBS) of a resin modified glass ionomer cement (RMGIC) to dentin.

Methods and Materials: Forty human third molar teeth were randomly divided into four groups (n=10). The occlusal enamel was removed to obtain a flat surface of dentin. Each group was treated as follows: Group 1: 10% polyacrylic acid (positive control); Group 2: 37% phosphoric acid followed by 5% sodium hypochlorite (NaOCl); Group 3: 1.1% APF gel; and Group 4: no conditioning (negative control). Fuji II LC glass ionomer was bonded to dentin using a cylindrical mold. Samples were thermocycled and debonded using a shear force with a crosshead speed of 0.5 mm/min. Data were analyzed using one-way analysis of variance (ANOVA) and Tukey tests ($\alpha = 0.05$).

Results: The mean SBS in Groups 1 through 4 were 11.562 ± 3.148 , 8.060 ± 1.781 , 8.830 ± 1.554 , and 3.074 ± 0.784 (MPa), respectively. There were significant differences in the SBS between Group 1 with other groups ($P < 0.05$). There were no statistically significant differences between Groups 2 and 3, but the SBS of both of them were significantly higher than that of Group 4 ($P < 0.05$).

Conclusion: Although the dentin SBS of Fuji II LC after conditioning with APF and phosphoric acid followed by NaOCl was greater than the unconditioned group (Group 4), polyacrylic acid yielded the best result.

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Clinical Significance: Proper conditioning of dentin is effective in promoting close adaptation of RMGIC to dentin.

Keywords: Shear bond strength, SBS, glass ionomer, surface conditioning

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Introduction

Resin-modified glass ionomer cements (RMGIC) represent a category of glass ionomer cement (GIC) modified by the addition of monomer components to create a hybrid material. These modifications offer improved physical properties.¹ RMGIC sets via two polymerization reactions: activation by light curing and by an acid-base chemical reaction.²

Czarnecka et al.³ claimed the chemical effects arising from interaction of carboxylic components of the cements and the calcium present in enamel and dentin substrates is the most important factor in the development of a strong bond. However, this adhesion mechanism is weak due to the presence of the smear layer which may interfere with adhesion.^{4,5} To overcome this drawback and enhance bonding, different surface treatment agents have been suggested to remove the smear layer prior to placement of GIC.⁵

Polyacrylic acid is the most commonly used conditioner for conventional GICs⁶ because it is capable of cleansing the dentin surface without completely unplugging the dentinal tubules.^{7,8} The increase in bonding efficiency resulting from conditioning can be attributed to the following:

- A cleansing effect which removes loose cutting debris following cavity preparation
- A partial demineralization effect which increases the surface area and creates microporosities
- A chemical interaction of the polyalkenoic acid with residual hydroxyapatite

The polyalkenoic acid pretreatment is much milder than a traditional phosphoric acid treatment, and the exposed collagen fibrils are not completely denuded of hydroxyapatite. A network of hydroxyapatite-coated collagen fibrils



interspersed with pores is typically exposed up to 1 μm in depth.⁹

Different concentrations of polyacrylic acid (25%, 20%, and 10%) in comparison with 12% citric acid or 4% aluminum chloride (AlCl_3) as surface treatment materials showed no differences in microtensile bond strength of Fuji II LC (GC Inc., Tokyo, Japan) to dentin. In addition, the microtensile bond strength of conditioned specimens of Fuji II LC was significantly greater than unconditioned specimens.¹⁰

A combination of 37% phosphoric acid followed by diamine silver fluoride and potassium iodide has also been suggested as a surface treatment which demonstrated similar dentin bond strengths to polyacrylic acid conditioners when bonding auto-cured glass ionomer.¹¹ The results of previous studies of the effect of dentin adhesives on the SBS of RMGIC to dentin demonstrated self-etching adhesives improved the bond strength of Fuji II LC to dentin.¹²⁻¹⁵

NaOCl is a non-specific proteolytic agent that effectively removes organic components at room temperature and has been frequently used for collagen removal with controversial results.¹⁶⁻¹⁸ While improvement in adhesion to deproteinized dentin with NaOCl has been reported, a few reports indicate interference by the chemical or a decrease in adhesion following its application.^{19,20} The decrease in bond strength after NaOCl application has been attributed either to the loss of the shock absorbing effect of the hybrid layer or the reduction in the physical properties of dentin.²¹

There are no studies in the literature that have evaluated the effect of acidulated phosphate fluoride (APF) as a surface conditioner on the dentin bond strength of RMGIC. Only one recent study found fluoride application during an initial dental prophylaxis had no effect on the bond strength values of Fuji Ortho LC, whereas it significantly lowered the bond strength values when applied before both conditioning and bonding.²²

Accordingly, the purpose of the present study was to assess the effect of different surface treatments on the shear bond strength (SBS) of a RMGIC (Fuji II LC) to dentin. The null hypothesis was: there are no significant differences in bond strengths among different surface treatment groups.

Methods and Materials

The tested materials with their compositions, specifications, and manufacturers are displayed in Table 1.

Forty caries-free extracted human third molars were selected and stored in 0.1% thymol-saline solution for one month following extraction. The teeth were individually positioned in a sectioning machine and a cut was made perpendicular to the longitudinal axis with a water cooled diamond saw (Isomat, Buehler Ltd, Lake Bluff, IL, USA) at low-speed to remove their occlusal surfaces to expose a flat, superficial dentin surface for testing (Figure 1).

To confirm the complete removal of enamel, the exposed dentin surfaces were viewed under an optical microscope. A standardized smear layer was produced on the dentin surface using #320 and #600 grit wet silicon carbide (Sic) paper in a APL 4 (Arotec, Coria, Sao Paulo, Brazil) polishing machine. Then adhesive vinyl tape with a 3 mm diameter hole was attached to the prepared dentin surface to demarcate and isolate the bonding site. The teeth were then randomly assigned to four groups of equal size (n=10), corresponding to type of surface treatments to be employed as follows:

Table 1. RMGIC, conditioners, compositions, specifications, and manufacturers.

Material	Type	Composition	Manufacturer
Fuji II LC	RMGIC	Powder: fluor-aluminosilicate glass Liquid: acrylic-maleic acid copolymer, 40% HEMA, water, comphorquinone	GC Inc., Tokyo, Japan
GC Dentin Conditioner	Acid solution	10% polyacrylic acid	GC Inc., Tokyo, Japan
Sodium Hypochlorite	Solution	5% NaOCl	
Phos-Flur Gel	Gel	1.1% acidulated phosphate fluoride (APF)	Colgate, New York, NY, USA
Scotchbond Etchant	Acid gel	37% phosphoric acid	3M ESPE, St. Paul, MN, USA

- **Group 1:** GC dentin conditioner (GC Corp, Tokyo, Japan) was applied to the dentin surface by scrubbing for 20 seconds, rinsed, and gently air dried (positive control).
- **Group 2:** Dentin surfaces were etched with a 37% phosphoric acid gel (Scotchbond Etchant, 3M ESPE, St. Paul, MN, USA) for 15 seconds, rinsed thoroughly, and gently dried. A 5% NaOCl solution was then applied on the etched dentin surface for 30 seconds, rinsed, and dried.
- **Group 3:** Surface treatment was performed using APF gel; Phos-Flur Gel (Colgate, New York, NY, USA) for one minute, then rinsed, and dried.
- **Group 4:** No surface treatment was done (negative control).

Fuji II LC RMGIC was mixed according to the manufacturer's instructions, loaded in a Centrix syringe, and inserted into a hydro-soluble gelatin mold (2 mm in height × 3 mm in diameter) then placed over the demarcated dentin site. The diameter of the mold was precisely aligned with the central hole created in the vinyl adhesive tape attached to the surfaces of the specimens. The material was light cured for 40 seconds on each side of the mold using an Astralis 3 light curing unit (Vivadent, Schaan, Liechtenstein). The mold was then removed, thereby, leaving RMGIC specimens adhered to the demarcated dentin sites.

The specimens were stored for 24 hours in deionized water at $37 \pm 1^\circ\text{C}$, then thermocycled (1000 cycles) in $5^\circ\text{C} \pm 2^\circ\text{C}$ and $55^\circ\text{C} \pm 2^\circ\text{C}$ with a dwell time of 45 seconds. After the vinyl tape was carefully removed, the specimens were placed in a universal testing machine (Zwick, Ulm, Germany) and loaded in shear at a crosshead speed of 0.5 mm/min (Figure 2).

The non-parametric Komogrov-Smirnov test accepted the normality hypothesis of data ($P > 0.05$); therefore, one-way analysis of variance (ANOVA) and Tukey Posts hoc tests were used to statistically analyze the SBS data at a confidence limit of 95%.

The debonded surfaces were examined with a stereomicroscope at 40X magnification to assess the failure modes which were classified as adhesive, cohesive, and mixed.



Figure 1. Schematic representation of the specimens.

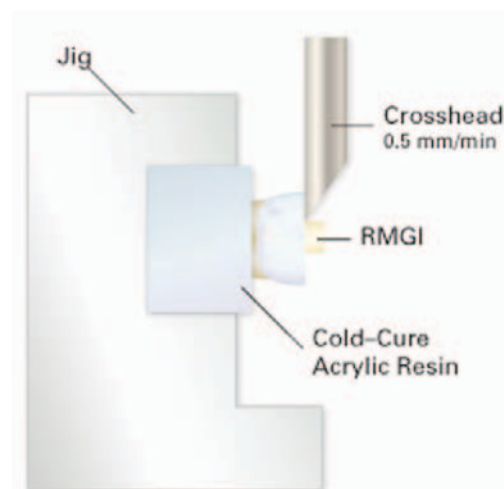


Figure 2. Schematic representation of the load application for measuring SBS.

Results

Table 2 shows the results of SBS (MPa) in the four groups. Group 1 using GC dentin conditioner (10% polyacrylic acid) yielded a significantly higher bond strength to dentin than the other three groups ($P < 0.05$). The obtained SBSs using sodium hypochlorite and Phos-Flur gel showed no significant differences ($P > 0.05$). The lowest SBS value was obtained in the unconditioned group (negative control).

The stereomicroscope observation of the fractured specimens revealed all treatment materials demonstrated an adhesive failure mode. Four specimens in the positive control group experienced mixed failures while cohesive failures were not observed.

Discussion

Since surface treatment is an essential clinical step to achieve improved bond strength, different materials were proposed in this study. The null hypothesis of this study was rejected because

Table 2. SBSs (MPa) of tested groups.

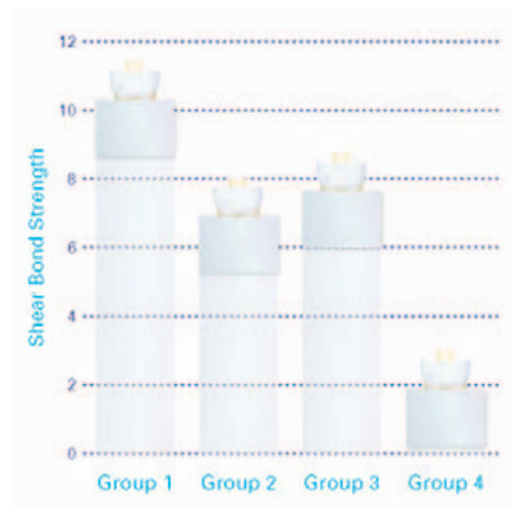
Material	N	Mean (MPa)	Standard Deviation
Group 1: GC Dentin Conditioner	10	11.562 ^a	3.148
Group 2: Scotchbond Etchant /NaOCl	10	8.060 ^b	1.781
Group 3: Phos-Flur Gel	10	8.830 ^b	1.554
Group 4: No surface treatment	10	3.074 ^c	0.784
Same letters show no significant difference (P>0.05)			

there were significant differences in the dentin bond strength of Fuji II LC following the use of different surface treatments. The bond strength obtained in this study ranged from 3.074 MPa to 11.562 MPa, while generally the bond strength of RMGIC ranges from 10 to 12 MPa.²³

Polyacrylic acid (11.562 MPa) yielded the best result which was significantly higher than the other groups in the study. This finding was consistent with previous studies which also demonstrated a significant improvement in the bond strength of Fuji II LC after conditioning with polyacrylic acid.^{7,10,13} Pereira et al.²⁴ observed resin tag formation in dentin specimens pretreated with polyacrylic acid and restored with Fuji II LC. Fuji II LC contains HEMA which can facilitate an improvement in the wetting ability as well as suitable bonding.²⁵ A hybrid-like layer was reported to form at the Fuji II LC/dentin interface when conditioning was carried out prior to application of this cement.¹⁰

The mean bond strengths of unconditioned specimens in the present study were significantly lower than all conditioned specimens. Contrary to the results of the present study, Terata et al.²⁶ and Inoue et al.²⁷ found no significant difference in the dentin bond strengths of RMGIC between the unconditioned group and specimens conditioned with 10% polyacrylic acid.

The SBS of NaOCl and APF to dentin were statistically similar (P>0.05), but Group 1 treated with polyacrylic acid (positive control) yielded a higher bond strength. There was no published



data on the effect of NaOCl as a treatment agent of dentin before applying the RMGIC.

NaOCl can dissolve proteins and reacts with the mineral phase of dentin. The treatment of dentin with NaOCl suggests a much slower process for deproteinization than for the demineralization using acid etching. The collagen fibrils within dentin are encapsulated by hydroxyapatite. After demineralization of dentin with phosphoric acid, the collagen exposed on the dentin surface is quickly attacked and removed by the sodium hypochlorite.²⁸

In the present study NaOCl was applied following the application of 37% phosphoric acid for surface treatment of dentin. NaOCl treatment probably led to a mineral surface rich in hydroxyl carbonate

and phosphate groups which become available for bonding. The results showed deproteination could increase the dentin SBS value of Fuji II LC compared with the unconditioned group.

A 5% NaOCl solution was applied to the dentin for 30 seconds in the present study based on findings of Perdigao et al.¹⁷ who used NaOCl for dentin deproteination using different application times. Their findings showed an increase in the NaOCl application time resulted in a progressive decrease in SBSs for dentin adhesives.

Some previous studies showed dentin deproteination by NaOCl eliminates the formation of the hybrid layer and establishes a higher bond strength of resin composite to dentin.^{17,20,29} In contrast, a decreased bond strength after NaOCl treatment has been reported for some bonding systems.^{30,31} Residual NaOCl and its oxygen by-product demonstrated a negative effect on the polymerization capacity of an adhesive system which caused reduction of bond strength. This effect is more profound when a total-etching system was used.³²

Sodium ascorbate has been used as a reducing agent that interacts with the oxygen by-product of NaOCl through a redox reaction, it is applied to NaOCl-treated dentin and rinsed away with water to achieve a normal bond strength.^{33,34} Several studies have shown sodium ascorbate can eliminate the oxidation effect of NaOCl resulting in a higher bond strength of composite to dentin and less microleakage.³⁵ In the present study application of sodium ascorbate following NaOCl was not done because the aim of the study was

to evaluate the effect of NaOCl as a surface treatment material on dentin.

This study demonstrated the dentin bond strength of Fuji II LC using APF as a surface treatment was statistically similar to phosphoric acid/NaOCl. Therefore, an APF-treated dentin surface was expected to show a greater propensity for RMGIC probably because of its chemical compatibility. APF is also easier to use than phosphoric acid/NaOCl. A search of the dental literature revealed no published studies on the effect of fluoride application on the bond strength values of RMGIC to dentin. A previous study has shown RMGIC can absorb fluoride from sources such as fluoride toothpastes and gels, thus, acting as a rechargeable system to produce a slow-release of fluoride.²² This phenomenon represents an additional benefit of using RMGIC as a restorative material in terms of recurrent caries prevention.

Further efforts need to be directed to improving the adhesive and bonding properties of filling materials placed on dentin.

Conclusion

Within the limitations of this study, polyacrylic acid treatment showed the highest bond strength. There was no statically significant difference between phosphoric acid/sodium hypochlorite treatments with regard to the Fuji II LC to dentin bond strength. The unconditioned group showed the lowest bond strength.

Clinical Significance

Conditioning of dentin is effective in promoting close adaptation of RMGIC to dentin.

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About the Authors

Hila Hajizadeh, DDS, MS



Dr. Hajizadeh is an Assistant Professor in the Department of Restorative Dentistry of the Mashhad Dental School and Dental Research Center at Mashhad University of Medical Sciences in Mashhad, Iran.

e-mail: hajizadehh@mums.ac.ir

Marjaneh Ghavamnasiri, DDS, MS



Dr. Ghavamnasiri is a Professor and the Director of the Postgraduate Program in the Department of Restorative Dentistry of the Mashhad Dental School and Dental Research Center at Mashhad University of Medical Sciences in Mashhad, Iran.

e-mail: ghavamnasirim@mums.ac.ir

Mohammad Sadegh Namazikhah, DMD, MSED



Dr. Namazikhah is a Professor and former Chair of the Department of Endodontics and Director of the Advanced Endodontic Program at the University of Southern California School of Dentistry in Los Angeles, CA, USA.

e-mail: namazikhah.sadegh@verizon.net

Sara Majidinia, DDS



Dr. Majidinia is a general dentist in private practice in Mashhad, Iran.

e-mail: sara_majidinia@yahoo.com

Mahshid Bagheri, DDS



Dr. Bagheri is a general dentist in private practice in Mashhad, Iran.

e-mail: mahshid1983@hotmail.com

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