

BOND STRENGTH OF RESIN-MODIFIED GLASS IONOMER TO DENTIN: THE EFFECT OF DENTIN SURFACE TREATMENT

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Abstract

Objectives: The purpose of this study was to evaluate the bond strength a resin-modified glass ionomer cements to dentin employing different dentin surface treatments.

Materials and Methods: Forty (40) sound and erupted thirds molars were selected and embedded in a 3/4 inch diameter PVC ring. The occlusal surfaces were ground using 180 and 320 grit Al₂O₃ abrasive paper with constant water irrigation until dentin was exposed. The specimens were randomly assigned to four groups (n = 10): G1- No dentin treatment; G2 - Dentin treated with 20% polyacrylic acid; G3 - Dentin treated with 37% phosphoric acid and left moist, and G4- Dentin treated with 37% phosphoric acid and dried. The resin-modified glass-ionomer cements, was used for restoration according to manufacturer's instructions. After 24 hours, specimens were tested for shear bond strength at 1mm/min crosshead speed. Data was evaluated by ANOVA and Fisher's test, at a 5% confidence level.

Results: The results [MPa (SD)] were as follows: G1=8.21(3.14), G2=11.30(1.24), G3= 8.74(1.96), G4= 10.36(1.18).

Conclusions: The treatment of dentin with 20% polyacrylic acid resulted in significantly higher bond strength values of Fuji II LC resin-modified glass-ionomer cement to dentin when compared to no dentin treatment or 37% phosphoric acid with moist dentin. The treatment of dentin with 20% polyacrylic acid showed a non-significant increase in bond strength values when compared to 37% phosphoric acid with dry dentin.

Clinical Significance: When bonding Fuji II LC to dentin, it is recommended that dentin is pre-treated with 20% polyacrylic acid or 37% phosphoric acid with dry dentin.

Key words: Bond strength; Resin-Modified Glass-Ionomer; Dentin; Superficial treatment

Short Title: Bond Strength of Fuji II LC to Dentin

Introduction

The continuous developments of new materials and techniques that promote effective adhesion between restorative materials and hard tooth structures have been the focus of many research groups.¹⁻⁴ The consistent bond strengths obtained in enamel substrate in 1955 by Buonocore⁵ after acid etching the enamel surface were not observed for dentin with the use of

first and second generation dentin adhesive systems. However with the introduction of the 3rd and subsequent generations of adhesive systems, satisfactory bond strength values have been observed for dentin.⁶ However, the bond strengths in dentin have not been as reliable as those found in enamel.⁷

The problems related to composite materials and adhesive systems have resulted in the indication for the use of glass-ionomer cements (GICs) as restorative materials in low-stress-bearing restorations and as a lining material. GICs exhibit several clinical advantages when compared to other restorative materials.⁶ They include physico-chemical bonding to tooth structures,^{2,8,9} fluoride release,³ and low coefficient of thermal expansion.^{10,11} Glass-ionomer's mechanism of bonding is based on bond formation between the carboxyl groups of polyacrylic acid with hydroxyapatite at the tooth surface.^{9,12,13}

Despite the chemical interactions between the GICs and dentin, the bond strength values of these materials continues to be low when compared to the bond strength of the adhesive systems to dentin and enamel after acid etching both

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Table 1 – Materials employed, composition and manufacture.

Materials employed	Composition	Manufacturer
Fuji II LC	Powder: fluoro-alumino-silicate glass Liquid: polyacrylic acid, 2-hydroxyethyl methacrylate (HEMA), dimethacrylate, camphorquinone, water	GC Corporation
Dentin conditioner	10% Polyacrylic acid, 10% aluminum chloride	GC Corporation
Dental conditioning	37% Phosphoric acid	Dentsply Caulk

substrates.^{7, 14} With the introduction of resin-modified glass-ionomer cements (RMGICs), which incorporate resinous matrix in its composition, there has been renewed interest in the adhesive ability of such restorative materials to tooth structure.

A chemical modification in the composition of GICs has resulted in several advantages of the material^{9, 15} and did not impair their critical adhesive ability and fluoride release properties.¹⁰ These properties make the RMGICs the first choice in cavities with high risk of secondary caries development.¹¹

Studies have shown that smear layer has been considered to be a major barrier in the bonding of RMGICs, particularly to dentine.^{1, 14-16} To optimize this bonding the treatment with polyacrylic acid can be considered as one of the first choices, however recently the phosphoric acid has been suggested for this purpose.^{4, 8, 9, 17} The use of phosphoric acid demands the wet dentine technique to avoid the collapse of the exposed collagen fibers and therefore a better diffusion of the hydrophilic monomers into the conditioned dentine surface. Yiu et al.⁹ have shown the presence of spherical bodies along the GICs-dentine interface when this material is applied in wet dentine, which has important implication on adhesive bonding.

Considering the importance of reliable bond strength values of restorative materials, the purpose of this study was to evaluate the bond strength values of RMGICs (Fuji II LC) to dentin, employing polyacrylic and phosphoric acid as dentin surface pre-treatments.

Materials and methods

Specimen preparation

For this study, forty (40) sound and erupted thirds molars were selected, cleaned of debris, and stored in distilled water until preparation. The study protocol was approved by the Human Subject Review Committee of the University of the State of São Paulo – Araçatuba School of Dentistry (Araçatuba, SP, Brazil).

In each tooth, the root was embedded in a 1,905 cm diameter PVC ring, with the occlusal surface parallel to the base of the ring. The rings were then filled with dental stone (Durone – Dentsply, Petrópolis RJ, Brazil) leaving the crown free.

The occlusal surface of the crowns were ground on a mechanical grinder (DCL - Dentária Campineira Ltd) using 180 grit Al₂O₃ abrasive paper (BUEHLER – Lake Bluff, IL, USA) with constant water irrigation until dentin was exposed. A guide apparatus was used during enamel grinding in order to standardize a flat occlusal surface perpendicular to the long axis of the tooth. Subsequently, 320 grit Al₂O₃ abrasive paper (BUEHLER – Lake Bluff, IL, USA) was used to obtain a flat dentin surface. To standardize the smear layer thickness, an apparatus was used over the grinding machine that allowed a constant pressure of 250 grams to grind the dentin. During all procedures the teeth were kept immersed in distilled water.

Bonding Procedure

Specimens were rinsed with tap water for 10 seconds and dried with oil free air spray. The specimens were randomly assigned to four groups (n = 10). Before the surface treatment, a 3.5 mm circular area was left uncovered as a bonding site by placing a piece of vinyl tape with a 3.5 mm diameter punched hole over the dentin.

The restoration of the specimens was done following a random sequence with the related materials in Table 1. The groups received the following treatments: Group 1 – Specimens were restored with RMGICs, Fuji II LC (GC Corporation, Tokyo, Japan) without prior dentin surface treatment; Group 2 - Dentin surface was treated with 10% polyacrylic acid (GC Dentin Conditioner – GC Corporation, Tokyo, Japan) for 15 seconds by active application using a microbrush. The dentin was rinsed with water for 10 seconds, air dried and restored in the same manner described for Group 1; Group 3 – Dentin surface was treated with 37% Phosphoric acid gel (Dentsply – Brasil, Petrópolis RJ, Brazil) for 20 seconds, rinsed for 20 seconds and blot dried with absorbent paper leaving the dentin moist but with no excess of water on the surface. The surface was restored in the same manner described for Group 1 and Group 4 – Dentin was treated in the same manner described for Group 3 except that prior to Fuji II LC (GC Corporation, Tokyo, Japan) placement, the etched

dentin was air dried for 20 seconds leaving a dry dentin surface.

A 3.5 mm diameter by 3 mm high bipartite Teflon ring mold was clamped to the dentin surface such that the mold was positioned over the treated dentin. The entire system was adapted into the apparatus to permit tight adaptation of the Teflon mold to the dentin surface.

The mold was filled with Fuji II LC (GC Corporation, Tokyo, Japan), using a syringe (C-R® Syringe Centrix™ Speed Slot) system and light-cured for 40 seconds (Ultralux EL - Dabi Atlante, Ribeirão Preto SP, Brazil). To ensure complete polymerization of the restorative material, after 5 minutes the teflon matrix was removed and the cylinder was additionally light cured for 40 seconds on each side of the cylinder, totaling 120 seconds. The light intensity was measured periodically by a radiometer (Demetron Research Corporation - USA), and averaged 450 mW/cm². Afterwards, specimens were immediately sealed with 2 coats of light-cured surface sealant (BisCover, Bisco, Schaumburg, IL, USA) to prevent either desiccation or water-sorption via external sources¹⁸, and were immersed in distilled water and stored at 37°C for 24 hours.

Shear bond strength test

Each specimen was mounted in a custom apparatus attached to a universal testing machine (EMIC Ltda, São José dos Pinhais, PR, Brazil) with the dentin surface parallel to the machine's trajectory. A compressive load was applied, using a steel knife-edge placed over the specimens, so that the force of the shear was applied directly to the bond interface. The specimens were

loaded to fail at a crosshead speed of 0.5mm/minute.

The data were subjected to One-Way analysis of variance (ANOVA) and Multiple Comparison Tukey's test with a 5% confidence level (α=0.05).

Results

The means and standard deviations of the values for each group tested are depicted in Table 02. After the detection of normal distribution of the samples, the values were subjected to One-way ANOVA, considering, dentin treatment, as being the only variable and Tukey's test, detected significant differences among the groups tested (p=0.00562). Groups 2 (Polyacrylic acid treatment) and 4 (Phosphoric acid treatment plus dried dentin) presented the highest values of bond strength with no statistically differences (p=0,3070). Groups 1 (no treatment) and 3 (Phosphoric acid treatment plus moist dentin), presented the lowest bond strength values and were not statistically significant different (p=0,5710). Although Group 4 presented high strength values they were not statistically different than Groups 1 (p=0,0244) and 3 (p=0,0840).

Discussion

Effective long term bonding of restorative materials to dentin substrate is the primary goal of many research groups. Intact restoration/dentin interfaces may indicate the ability of different materials to prevent recurrent caries development and post operative sensitivity as a result of microleakage at the interface.

Table 2 – Bond strength mean values and standard deviations of the groups tested.		
GROUPS	MEANS (Standard Deviation)	DECISION*
G1 (no treatment + Fuji II LC)	8.21 (3.14)	b
G2 (10% polyacrylic acid + Fuji II LC)	11.30 (1.24)	a
G3 (37% Phosphoric acid/moist dentin + Fuji II LC)	8.74 (1.96)	b
G4 (37% Phosphoric acid/ dry dentin + Fuji II LC)	10.36 (1.18)	ab

Tukey's critical values ==> t=2.458

* - Different letters indicate statistically significant differences between groups at 5% confidence level.

The use of adhesive restorative materials that offer good sealing ability combined with possible fluoride release could reduce and/or prevent complications related to the presence of marginal infiltration. GICs are potential materials to be placed in critical areas to obtain adhesion due to its chemical interaction with the underlying dentin.^{6, 15, 19} GICs restorative materials can promote optimal sealing and consequently protect against marginal infiltration. Its fluoride releasing ability may help to control recurrent caries development and pulpal pathology that could compromise restorative treatment in a short period of time.^{7, 20}

Following cavity preparation, a smear layer is formed on the surface of dentin. Studies^{1, 2, 14} have shown that this layer can impede the intimate contact of glass- ionomer material to dentin and consequently compromise the chemical and/or physical (micromechanical) interaction. This was confirmed in the present study, in which the lowest bond strength values were observed when dentin did not receive any treatment prior to Fuji II LC application (Group 1). On the other hand, acid treatment of dentin was able to optimize the bond strength values when compared to no dentin treatment, as it was observed in resin-modified glass-ionomers by De Munck et al.²¹ Smear layer removal by polyacrylic acid (Group 2) or phosphoric acid allowed for better interaction of the material with dentin and therefore increased bond strength values, demonstrating that the self-adhesiveness of RMGIs should be attributed to ionic bonding to hydroxyapatite around collagen, and to micro-mechanical interlocking for those RMGIs that additionally hybridize dentin.²²

The results of the present study show that dentin treatment with 10% polyacrylic acid resulted in the highest bond strength values, which was reported in other studies.^{4, 15, 16} Pretreatment with a diluted polyacrylic acid conditioner is recommended^{3, 12, 14} and has the ability to remove the smear layer, leave the smear plug and slightly partially demineralize dentin, leaving hydroxyapatite around exposed collagen fibrils^{8, 13, 23} accessible for interaction. This condition is favorable for chemical interaction of the carboxylic groups from Fuji II LC and hydroxyapatite crystals from dentin^{1, 7, 16, 24} and mechanical bonding via hybrid layer formation between dentin and RMGICs.^{13, 17} Consequently, polyacrylic acid-pretreatment is still to recommended in order to achieve more consistent and durable bonding of resin-modified glass-ionomers to dentin.²¹

Conversely, 37% phosphoric acid, used in Groups 3 and 4, removes the smear layer, smear plug and also promotes superficial demineralization of the dentin.^{13, 25} De Munck et al.⁸ recently reported that the pretreatment of dentin with 37.5% Phosphoric acid or 10% polyacrylic acid resulted in similar bond strength values, however, the authors do not address the moisture condition of the dentin. The phosphoric acid treatment promotes mineral removal from dentin, which is

important for chemical interaction with Fuji II LC, while at the same time allowing for mechanical interaction with the dentin provided through hybrid layer and tag formation (Figures 1, 2 and 3).

In Group 4, where after phosphoric acid application, dentin was air dried for 20 seconds, satisfactory bond strength values



Figure 1 - Histologic section of sound dentin, presenting Hybrid Layer (HL), Tags (T) – Fuji II LC. Material - Brown & Brenn Staining – Polarized Light Microscopic – 400x. Sundfeld et al. ¹⁷

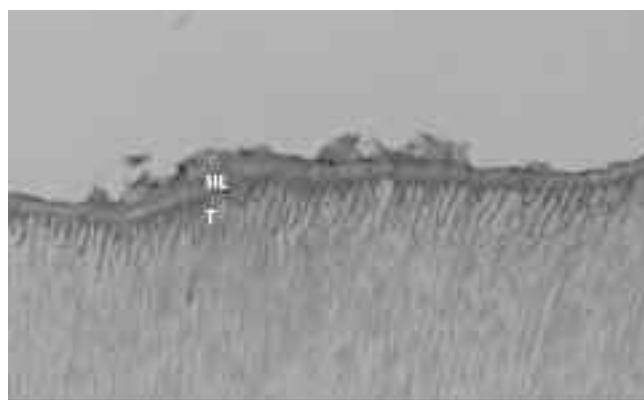


Figure 2 - Histologic section of sound dentin, presenting Hybrid Layer (HL), Tags (T) – Fuji II LC. Material - Brown & Brenn Staining – Polarized Light. Microscopic – 400x. Photographic effects SIGMA SCAN 4.0 program (FIT-1X-TRANSFORMS GRAY FILTERS - IM). Sundfeld et al. ¹⁷

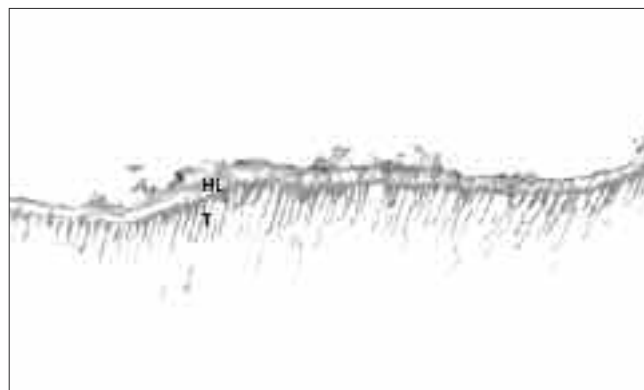


Figure 3 - Histologic section of sound dentin, presenting Hybrid Layer (HL), Tags (T) – Fuji II LC. Material - Brown & Brenn Staining – Polarized Light Microscopic – 400x. Photographic effects SIGMA SCAN 4.0 (FIT -1X-TRANSFORMS CONVERT TO GRAY SCALE - CGS). Sundfeld et al. ¹⁷

were observed. We suggest that due to the resinous composition (HEMA) of Fuji II LC, dentin tags were created and also a hybrid layer was formed, which are responsible for the bond strength values of resin based materials.^{17, 26} However, in Group 3 which was also pre-treated with phosphoric acid, but the dentin was left moist, lower bond strength values were observed when compared to Group 4. The results do not confirm the hypothesis reported by Yiu et al.⁹ who reported the presence of spherical bodies along the RMGICs/dentine interface. According to Yiu et al.,⁹ it is hypothesized that the spherical bodies, similarly to the adhesive layer, may serve to deflect or blunt any cracks that attempt to propagate through the matrix, thereby toughening the material. The spherical bodies may play an adjunctive role by obliterating porosities in the resin matrix adjacent to the dentine and delay the growth of inherent cracks to catastrophic sizes in this region under loading.^{14, 15} The present study suggests that although RMGICs are considered hydrophilic, they did not perform well in the presence of moisture, resulting in impaired chemical/physical interactions between the demineralized and moist dentin.^{4, 15}

Based on the results observed in the present study, it is important to recommend the use of restorative techniques and

materials that provide reliable bond strength values to dentin. Therefore, the application of 10% polyacrylic acid on dentin prior to placement of Fuji II LC RMGICs should be the first technique choice to be employed. This treatment showed the highest bond strength values and is also a mild acid composed of large molecules as compared to phosphoric acid and thereby is more biocompatible. In addition, the dentin pre-treatment using polyacrylic exposes a limited amount of collagen fibers that may be rapidly and fully encapsulated by the hydrophilic monomers of RMGICs and therefore less technique sensitive when compared to the phosphoric acid pre-treatment. Considering the possibility of adhesive degradation in wet dentin, along the time,^{9, 21, 22, 27, 28} the application of 37% phosphoric acid should be the second choice for acid pre-treatment of dentin, as long as the dentin is kept dry prior to Fuji II LC placement.

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