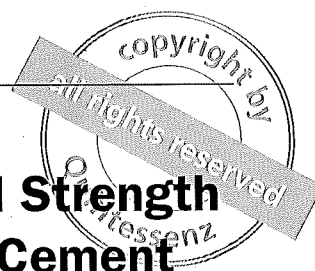




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Effect of Conditioning and Aging on the Bond Strength and Interfacial Morphology of Glass-ionomer Cement Bonded to Dentin

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Purpose: To determine the bond stability and the change in interfacial ultrastructure of a conventional glass-ionomer cement bonded to dentin, with and without pretreatment using a polyalkenoic acid conditioner.

Materials and Methods: The occlusal dentin surfaces of 10 teeth were ground flat. Glass-ionomer cement was bonded to the surfaces either with or without polyalkenoic acid conditioning. The teeth were sectioned into 1-mm² stick-shaped specimens. The 200 specimens obtained were randomly assigned to four groups with different periods of storage in water: 1 week, 1 month, 3 months, and 6 months. The microtensile bond strength (μ TBS) was determined for each storage time. Additional specimens were prepared for transmission electron microscopy (TEM); they were produced with or without prior polyalkenoic acid conditioning in the same manner as for the μ TBS test.

Results: There was no significant difference in μ TBS to conditioned dentin ($p > 0.05$). After 6 months of aging, the μ TBS to non-conditioned dentin was significantly reduced as compared to the 1-week, 1-month, and 3-month results ($p < 0.05$). The failures appeared to be of a mixed nature, although aging caused more areas of cohesive than adhesive failure in all groups. TEM observation showed a demineralized layer and an amorphous gel phase in the polyalkenoic acid conditioned group.

Conclusion: Aging did not reduce the bond strength of the conventional glass-ionomer cement to dentin when the surface was pretreated with a polyalkenoic acid conditioner.

Keywords: adhesion, dentin, glass-ionomer cement, microtensile bond strength, transmission electron microscopy.

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Conventional glass-ionomer cement (GIC) and resin-modified GIC (RMGIC) are currently the only materials that do not require extra provisions for consistent retention or adhesion, as they adhere directly to dental hard tissues even in a moist environment. RMGIC is characterized by the addition of photo-activated methacrylate and a small amount of resin to conventional GIC. Conventional GIC is commonly used in the Atraumatic Restorative Treatment (ART) technique in developing countries.⁶ Conventional GIC does not require light curing

and can bond chemically to hydroxyapatite (HAp) and has a less demanding technique than resin-based restorations. Furthermore, GIC has a pH-buffering capacity, as it releases fluoride proportionally to the acidity^{4,13} and is also biocompatible.^{15,21}

While the resin/tooth interface may degrade over time, the GIC material itself undergoes a maturation effect, which possibly improves the durability of its bond to dentin.¹⁴ Clinically, the performance of conventional GIC in non-retentive cervical lesions is favorable, even up to five

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**Table 1** The materials used in this study

PRODUCT NAME	COMPOSITION
Cavity Conditioner (GC,Tokyo, Japan)	20% polyalkenoic acid, distilled water, 3% aluminum chloride, food additive Blue No. 1
Fuji IX GP Extra (GC)	Polyacrylic acid, aluminosilicate glass, proprietary ingredient
GC Fuji Coat LC (GC)	Multifunctional urethane methacrylate, aliphatic dimethacrylate, methyl methacrylate, tertiary amine

years, and not significantly worse than that of composite resins bonded with the clinical "gold-standard" three-step etch-and-rinse adhesives.^{18,25} Due to its favorable performance in some clinical trials,^{18,25} the interface between teeth and GIC has received renewed interest. GIC demineralizes dentin only superficially, leaving HAP around exposed collagen fibrils, which provides micromechanical retention.¹⁰ The residual HAP serves as a receptor for additional chemical bonding, involving ionic interaction of the carboxyl functional groups of polyalkenoic acid with calcium remaining at the interface.^{9,10} To maximize chemical bonding, pretreatment with a polyalkenoic acid conditioner is often recommended. Some laboratory studies have reported improvement in bond strength after surface pretreatment (conditioning) with various solutions.^{1,2,8,12,19} In contrast, other studies have found that certain GICs adhere to tooth structure without prior treatment.^{7,23} It is still unclear whether the conditioning of GIC is effective. Moreover, little information is available on the long-term durability of GIC in terms of bond strength and interfacial ultramorphology.

The purpose of this study was to assess the bond durability of a conventional GIC to dentin over time, with or without pretreatment using a polyalkenoic acid conditioner. The null hypothesis tested was that pretreatment using a polyalkenoic acid conditioner did not affect the bond durability of a conventional GIC. To test this hypothesis, a microtensile bond strength (μ TBS) test and transmission electron microscopy (TEM) interfacial analysis were used to correlate the bond strength with ultrastructural information.

MATERIALS AND METHODS

Microtensile Bond Strength (μ TBS)

The bond strength to dentin was determined using a standard microtensile bond strength test.²⁰ The materials used in this study are shown in the Table 1. Ten human third molars, stored in a 0.5% chloramine T solution, were used within 1 month of extraction. The protocol of this research was approved by the Commission for Medical Ethics of KU Leuven. The extracted molars were sectioned at the mid-coronal portion using a low-speed diamond saw (Isomet 1000, Buehler; Lake Bluff, IL, USA) to create a flat dentin surface. A standard smear layer was produced using a Micro-Specimen Former (University of Iowa; Iowa City, IA, USA) equipped with a high-speed regular-grit (100- μ m)

diamond bur (842, Komet; Lemgo, Germany). The teeth were randomly divided into two groups of 5 teeth each. Prior to the application of the GIC, the dentin surface of the specimens in one group was pretreated with a polyalkenoic acid conditioner (Cavity Conditioner, GC; Tokyo, Japan). This contains 3% aluminum chloride as well as 20% polyalkenoic acid. The specimens in the other group did not receive any pretreatment. The dentin surface was subsequently built up freehand and in bulk with a conventional GIC (Fuji IX GP Extra, GC) to a height of 5 to 6 mm, followed by application of a surface sealer (GC Fuji Coat LC, GC) and light cured for 10 s.

After 1 week of storage in distilled water at 37°C, the specimens were sectioned perpendicular to the bonding surface to obtain 1-mm² stick-shaped microspecimens using an automatic precision water-cooled cutting machine (Accutom-50, Struers; Ballerup, Denmark). The specimens were randomly assigned to 8 groups (25 specimens each) according to age/storage time: 1 week, 1 month, 3 months, and 6 months, ie, the 1-week specimens were tested after sectioning while the rest remained in storage up to a total of 1 month, 3 months, or 6 months. At the relevant time period, the microspecimens were fixed to a jig with cyanoacrylate glue (Model Repair II Blue, Dentsply-Sankin; Ohtawara, Japan) and stressed in an LRX testing device (Lloyd; Hampshire, UK) at a crosshead speed of 1 mm/min until failure occurred. The μ TBS was calculated in MPa, derived by dividing the force applied (in N) at the time of fracture by the bonded area (in mm²). Statistical analysis was performed using one-way ANOVA ($\alpha = 0.05$) and post-hoc Tukey-Kramer multiple comparisons tests. The mode of failure was determined at a magnification of 50X using a stereomicroscope (Wild M5A; Heerbrugg, Switzerland).

SEM Failure Analysis

Two representative samples of each group were prepared for scanning electron microscopy (SEM) examination using standard specimen processing techniques (glutaraldehyde fixation, gradual dehydration, hexamethyldisiloxane drying). The specimens were mounted on stubs and coated with a thin gold layer using an automatic sputter coater (JFC-1300, JEOL; Tokyo, Japan) before being examined using the SEM (JSM-6610 LV, JEOL).

TEM Interface Characterization

Additional specimens were prepared for examination using TEM (JEM-1200EX II, JEOL). For this, a further 8 teeth were randomly divided into 2 groups of 4 teeth

each; the dentin was pretreated with polyalkenoic acid conditioner in one group but not in the other. The procedure of bonding the GIC to dentin was the same as previously described in the μ TBS test before storage in distilled water for 1 week and 6 months at 37°C. After storage for each time period, the GIC-bonded dentin specimens were sectioned perpendicular to the GIC/dentin interface using an Isomet diamond saw (Buehler). From each tooth, seven or eight rectangular sections were obtained. TEM sample preparation was performed in accordance with common procedures used for ultrastructural TEM examination of biological tissues.²⁷ This involved fixation overnight in 2.5% glutaraldehyde in 0.1 M sodium cacodylate buffer at pH 7.4 and 4°C, followed by rinsing in 0.1 M sodium cacodylate buffer for 1 min with 3 changes. Dehydration was carried out in ascending grades of ethanol (50%, 75%, 95%, 100%) for 10 min each, with 2 changes. This was followed by immersion in 1:1 absolute ethanol-epoxy embedding resin for 30 min, and then resin infiltration in 100% epoxy embedding resin for another 4 h. Finally, embedding of the resin-infiltrated samples in molds with 100% epoxy resin was carried out. Before being embedded, the specimens were oriented in the molds so that ultrathin sections through the GIC/dentin interface could be cut from the dentin part of each original tooth. The epoxy blocks were polymerized in an oven at 60°C for a minimum of 48 h. Subsequently, non-demineralized, 70- to 90-nm-thick sections were cut using a diamond knife (Diatome; Bienne, Switzerland) in an ultramicrotome (Ultracut UCT, Leica; Vienna, Austria). The GIC/dentin interface in each section was observed by TEM at magnifications of 25,000X and 50,000X.

RESULTS

Microtensile Bond Strength (μ TBS)

The mean μ TBS values are presented in Fig 1. Any pre-test failures (ptf) were considered as 0 MPa (the number of ptf in each group is indicated in Fig 1). There was no significant difference in μ TBS to conditioned dentin at various time periods ($p > 0.05$). Up to 3 months of aging, there was no significant difference in μ TBS to non-conditioned dentin ($p > 0.05$), while 6 months' aging significantly reduced the μ TBS to non-conditioned dentin as compared to the 1-week, 1-month, and 3-month results ($p < 0.05$). For each time frame, there were no significant differences between conditioned and non-conditioned dentin ($p > 0.05$).

SEM Failure Analysis

At 1 week, the failure patterns were generally of a mixed nature, involving areas that failed at the interface and areas that failed cohesively within the GIC, for both the conditioned and non-conditioned groups. However, these mixed failures mostly involved areas with interfacial failure between dentin and GIC, more than areas with cohesive failure within the GIC. At other time periods, while failure was still mixed, there was a tendency

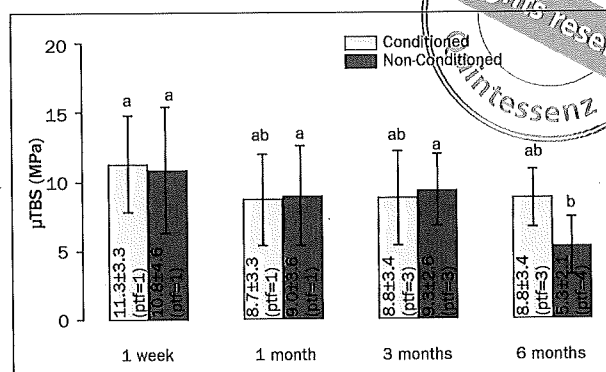


Fig 1 Microtensile bond strength of GIC bonded to polyalkenoic acid conditioned (Cavity Conditioner) and non-conditioned dentin for the four different storage periods. Mean μ TBS in MPa ($n = 50$). Same letters indicate no statistically significant difference ($p > 0.05$). Any pre-test failures (ptfs) were considered as 0 MPa.

for more areas of cohesive failure with the passage of time. Hence, aging of both conditioned and non-conditioned specimens caused them to fail slightly more frequently cohesively within the GIC.

TEM Interface Characterization

Representative TEM photomicrographs of unstained, non-demineralized sections of the GIC/dentin interface of specimens stored for 1 week are shown in Fig 2, with (Figs 2a and 2b) and without (Figs 2c and 2d) polyalkenoic acid conditioning using Cavity Conditioner. A shallow, partially demineralized dentin layer of about 0.5 to 1 μ m was seen at the dentin-conditioned interface (Figs 2a and 2b). Hydroxyapatite (HAp) remained within this partially demineralized layer. On top of the partially demineralized layer, a gel-like zone of a few hundred nanometers was deposited, and this zone was typically demarcated from the GIC matrix by small electron-lucent globules (Figs 2a and 2b). When the dentin surface was not conditioned prior to placement of the GIC, TEM examination revealed that the GIC was closely attached to the dentin surface and no gel phase could be detected (Figs 2c and 2d).

Representative TEM photomicrographs of unstained, non-demineralized sections of the GIC/dentin interface in specimens stored for 6 months are shown in Fig 3, with (Figs 3a and 3b) and without (Figs 3c and 3d) polyalkenoic acid conditioning. As in Fig 2, a shallow, partially demineralized dentin layer of about 0.5 to 1 μ m was seen at the dentin-conditioned interface (Figs 3a and 3b). On top of the demineralized layer, a gel phase of a few hundred nanometers could be detected (Figs 3a and 3b). When the dentin was not conditioned prior to placement of the GIC, TEM examination revealed that the GIC closely attached to the dentin surface without a smear layer (Figs 3c and 3d). The bond appeared intact. No clear signs of bond degradation were observed after 6 months of water storage.

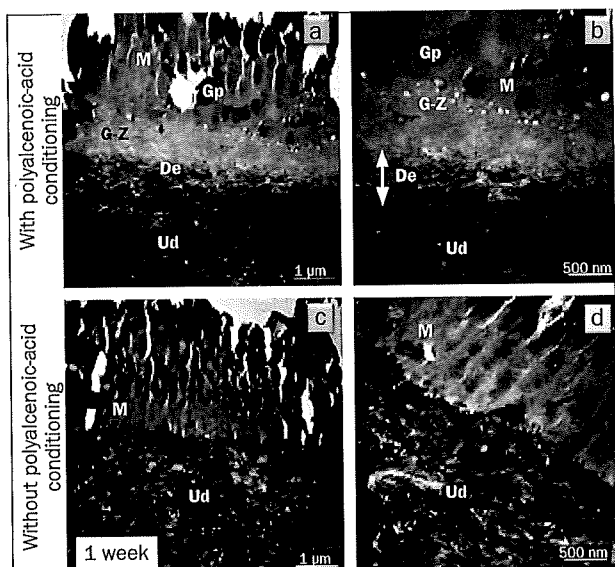


Fig 2 Representative TEM photomicrographs of unstained, non-demineralized sections of the GIC/dentin interface after storage for 1 week, with (a,b) and without (c,d) polyalkenoic acid conditioning using Cavity Conditioner. (a,b) A shallow partially demineralized dentin layer (De) of about 0.5 to 1 μ m resulted from polyalkenoic acid conditioning; the width of this layer is indicated by the arrowed line in Fig 2b. On top of this layer, a gray gel-like zone (G-Z) of just a few hundred nanometers thickness can be observed. This layer does not contain HAP or filler, and is typically demarcated from the glass-ionomer matrix (M) by small electron-lucent globules. (c,d) In contrast, when the GIC was bonded to non-polyalkenoic acid conditioned dentin, the material was closely attached to the dentin surface, where no demineralization is clearly seen. A gel-like zone could not be detected.

Ud = unaffected dentin; De = demineralized layer; G-Z = gel-like zone; Gp = glass particle in glass-ionomer; M = matrix in glass-ionomer. Figs 2a and 2c 25,000X magnification; Figs 2b and 2d 50,000X.

DISCUSSION

In this study, the long-term durability and interfacial ultrastructure of GIC/dentin bonds was studied using a μ TBS test and TEM, respectively. From the μ TBS test, the bond strength appeared to have significantly decreased after 6 months of aging when the dentin was not pretreated with a polyalkenoic acid conditioner. Some studies reported that there were no significant differences in bond strength of GIC bonded either to conditioned or non-conditioned dentin without aging.^{7,23} These studies were relatively short term regarding the debonding stress imposed. Yet other studies used RMGIC and did not assess conventional GIC.^{2,8,11} Information from these short-term studies suggest that micromechanical interlocking may confer resistance against short-term debonding stress. Additional chemical bonding has been shown to provide more durability to the interface over the long term.⁵ One of the reasons for applying polyalkenoic acid con-

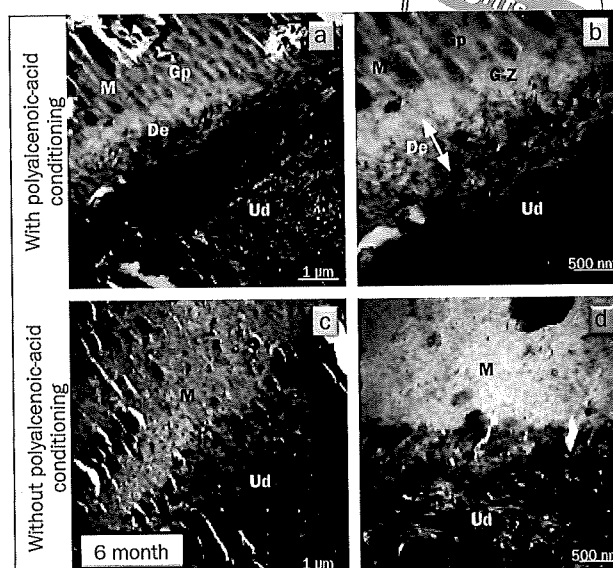


Fig 3 Representative TEM photomicrographs of unstained, non-demineralized sections of the GIC/dentin interface stored for 6 months with (a,b) and without (c,d) polyalkenoic acid conditioning using Cavity Conditioner. (a,b) A shallow demineralized layer (De) of about 0.5 to 1 μ m resulted from conditioning. (b) On top of this layer, a gray gel-like zone of a few hundred nanometers (G-Z) could be detected. (c,d) TEM revealed that when the GIC was bonded to non-conditioned dentin, it was closely attached to the dentin surface, where no demineralization is clearly seen. A gel-like zone could not be detected.

Ud = unaffected dentin; De = demineralized layer; G-Z = gel-like zone; Gp = glass particle in glass-ionomer; M = matrix in glass-ionomer. Figs 3a and 3c 25,000X magnification; Figs 3b and 3d 50,000X.

ditioner is to effectively deal with surface smear and to maximize the chemical bonding of GIC to dentin. In addition, polyalkenoic acid is recommended due to its high molecular weight (MW) and because it forms insoluble calcium salts.¹⁹ This may explain the difference in bonding effectiveness recorded in this study between conditioned and non-conditioned specimens in the long term.

No differences in failure pattern were observed between conditioned and non-conditioned specimens in the present study. Although the mode of failure was of a mixed nature, the primarily adhesive failure at the earlier time period was not maintained; more areas of cohesive failure within the cement itself were noted with aging (in both conditioned and non-conditioned specimens). Cohesive failure in GIC has been reported previously.^{23,24} The tendency of cohesive failure in this study may be due to numerous porosities, which may act as more stress points within the cement for cohesive failure to occur with time.

To date, the interaction of GIC with dentin has been less frequently documented by high-resolution microscopy, compared to resin-based adhesive interfaces. TEM analysis in this study revealed that GIC interacted with dentin following two distinct patterns. When GIC was bonded to conditioned dentin, a submicrometer partially demineralized layer was produced; HAp remained attached to collagen within this partially demineralized dentin layer. The deposition of the submicrometer gel phase over this layer has been previously interpreted as the morphological manifestation of a gelation reaction of the high-MW polyalkenoic acid with calcium that was extracted from the underlying dentin surface of RMGICs.³ When the GIC was applied without polyalkenoic acid conditioning, dentin was minimally demineralized. Hence, micromechanical bonding may be limited to retention provided by the intrinsic surface roughness of dentin and by the porous surface created by the material's self-etching characteristics. In addition, chemical bonding may occur, as was confirmed by a previous XPS study.³ This was also demonstrated by the μ TBS results, as the GIC applied without prior polyalkenoic acid conditioning did not reveal significantly different bond strength from the conditioned dentin despite the limited micromechanical interlocking at up to 3 months of aging. However, the relevance of the gel phase with regard to bond durability is not known, as is whether this gel phase and the partially demineralized dentin layer underneath are beneficial for long-term bond durability. The polyalkenoic acid conditioner produces this gel phase, as the carboxyl functional groups from polyalkenoic acid interact ionically with calcium released from the dentin surface. This would not occur when dentin is conditioned with phosphoric acid, as phosphoric acid would be rinsed off, thereby removing all HAp and calcium; if the surface is not rinsed, the calcium phosphates formed would be too soluble to form a gel.²⁹

Some studies have shown that GIC stored in saliva improved surface characteristics compared to GIC stored in distilled water.^{16,17,22,26} GIC may absorb some inorganic ions from saliva and this may increase the surface hardness over time. It would be worthwhile to assess the effect of GIC/dentin bond aging within saliva in future research.

Polyalkenoic acid has been recommended for conditioning dentin prior to the application of GICs. This instruction is based on the capacity of polyalkenoic acid to interact with HAp without forming soluble complexes. Previous x-ray photoelectron spectroscopy showed a shift of the -COOH peak to a lower binding energy when polyalkenoic acid was applied to HAp.²⁸ This represents the formation of ionic bonds between the carboxyl functional groups with calcium at the HAp surface.²⁸ It was previously demonstrated that the stability of the bond to HAp tends to depend on the solubility of the calcium salt formed during conditioning.²⁹

The results of the μ TBS test showed that pretreatment of dentin using a polyalkenoic acid conditioner affected the long-term durability of a conventional GIC; hence, the null hypothesis should be rejected.

CONCLUSION

Bond durability can be significantly improved by pretreating dentin using a polyalkenoic acid. Aging did not reduce the bond strength of the conventional GIC to dentin when the surface was pretreated with a polyalkenoic acid conditioner up to 6 months. Hence, the use of a polyalkenoic acid conditioner is clinically recommended.

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Clinical relevance: Aging did not reduce the bond strength of the conventional glass-ionomer cement to dentin when the surface was pretreated with a polyalkenoic acid conditioner. Conditioning of dentin appears to increase the durability of the glass-ionomer bond to dentin.