Effect of Chlorhexidine Application on the Long-term Shear Bond Strength to Dentin of a Resin-modified Glass Ionomer

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Clinical Relevance
The application of 0.05% chlorhexidine digluconate after polyalkenoic acid conditioning (Cavity Conditioner®) of dentin and prior to application of resin-modified glass ionomer cement (Fuji II LC®) should be avoided.

SUMMARY
Purpose: The aim of this study was to investigate the effect of chlorhexidine digluconate (CHX) application on the shear bond strength (SBS) of a resin-modified glass ionomer cement (RMGIC) to polyalkenoic acid-preconditioned dentin after 24 hours, six months, and 12 months of water storage at 37°C.

Materials and Methods: Cylindrical molds, placed on flat, polyalkenoic acid (Cavity Conditioner® [GC]) preconditioned dentin surfaces of 90 human teeth embedded in resin, were filled with Fuji II LC® (GC), a RMGIC, with (n=45) or without (n=45) the prior application of a 0.05% CHX solution. Within each group, SBS was determined after 24 hours (n=15), six months (n=15), and 12 months (n=15) of storage in water at 37°C. The results were analyzed with two-way analysis of variance followed by Tukey multiple means comparisons (p<0.05). The type of bond failure (adhesive/cohesive/mixed) was noted and the results were analyzed with chi-square test (p<0.05).

Results: After 24 hours, the SBS of RMGIC was not significantly different with (9.0 ± 2.8 MPa) or without (8.3±0.6 MPa) the application

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of CHX. After six months, however, SBS increased significantly in the group without CHX (12.7±3.4 MPa) but remained unchanged in the CHX group (9.4±4.0 MPa). Similar results without CHX (12.6±3.8 MPa) and with CHX (9.5±3.2 MPa) were obtained after 12 months. No significant differences in the type of debonding were found between the various groups tested.

Conclusion: The application of 0.05% CHX after dentin preconditioning did not seem to have affected the 24-hour SBS of RMGIC. However, the six- and 12-month SBS was significantly lower for CHX-treated samples, possibly as a result of CHX interference with both the bonding mechanism and the maturation reaction of RMGIC.

INTRODUCTION

The loss of bond strength is the major shortcoming that affects adhesive restorations and decreases their longevity. To prolong the clinical lifetime of adhesive restorations, it would be interesting to improve the stability of both the tooth tissue and of the involved adhesive interfaces.

Several authors have shown the hydrolytic degradation of collagen matrices in aged dentin-resin bonds, even in the absence of bacterial enzymes, via host-derived matrix metalloproteinases (MMPs), a class of zinc- and calcium-dependent endopeptidases, responsible for degrading practically all extracellular matrix components of connective tissues. Human dentin contains at least collagenase (MMP-8), gelatinases MMP-2 and MMP-9, and enamelysin MMP-20.

Studies have revealed that chlorhexidine (CHX) could function as a potent MMP inhibitor. With etch-and-rinse adhesive systems, pretreating the cavity with CHX after phosphoric acid-etching may prevent or delay the interfacial degradation of the dentin-resin bond, preserving the bond strength of in vitro aged specimens. With self-etching adhesives, some studies showed that CHX was able to diminish the loss of microtensile bond strength over time. However, with a self-adhesive resin cement, CHX seemed to have no effect on bonding durability.

Glass ionomer cements (GICs) and resin-modified glass ionomer cements (RMGICs) are materials that self-adhere to hard tooth tissues. A short polyalkenoic-acid pretreatment is recommended to clean the tooth surface, to remove the smear layer, and to expose collagen fibrils up to approximately 0.5-1-μm depth into which cement components could interdiffuse and establish a micromechanical bond, following the principle of hybridization, even if the chemical adhesion mechanism through ionic attraction is preponderant.

Previous studies have reported that CHX did not significantly affect the bond strength of RMGIC when CHX was applied before the dentin conditioner as a cavity disinfectant. To our knowledge, no studies have evaluated the effect of CHX applied after dentin conditioning on the bond strength of RMGIC to dentin. The aim of this study was to investigate the in vitro effect of 0.05% CHX on the shear bond strength (SBS) of a RMGIC applied to dentin preconditioned with polyacrylic acid after 24 hours, six months, and 12 months of storage in water at 37°C.

The null hypothesis tested was that CHX application after dentin conditioning has no effect on SBS over 24 hours, six months, and 12 months of ageing.

MATERIALS AND METHODS

Ninety freshly extracted human molars were collected, cleaned of soft tissue, stored in a solution of 1% chloramine-T at 4°C, and used within one month. The criteria for tooth selection included absence of cracks caused by extraction forceps as well as absence of decay. The teeth had been extracted for reasons unrelated to the objectives of this study and with the patients’ informed consent. The project was approved by the scientific council of the Faculty of Dental Surgery, University of Paris–Descartes. These selected teeth had the greater portion of the roots removed with use of sandpaper (80 grit). The occlusal surface of the crowns was then abraded on water-cooled sandpaper (800 grit) using a polishing machine (Planopols, Struers, Copenhagen, Denmark) to expose a flat dentin surface (7× mm ), onto which a cylinder of RMGIC could be formed and bonded. Finally, the residual crowns were embedded in self-cured acrylic resin in plastic cylinders with the flat dentin surface exposed. The flat surfaces were inspected under 40X magnification to ensure that the enamel had been completely removed and the dentin cleared of debris.

For all of the samples (n=90), polyalkenoic acid (Cavity Conditioner® [GC]) was applied onto the dentin surface, was left undisturbed for 10 seconds, was rinsed with water for 10 seconds, and was then gentle air-dried for five seconds to leave a moist surface.
Randomly, half of the samples (n = 45) were treated with 0.05% CHX (applied with a microbrush and blot-dried after a dwell time of 60 seconds) prior to bonding, while the other half (n = 45) were not. The 0.05% CHX concentration is lower than the 0.2% or 2% typically used, but it is a concentration commonly used in the clinic and sufficient to prevent interface degradation for up to six months.31

A cylindrical Teflon mold that allowed us to build cylinders of 2-mm height and a plane base of 3-mm diameter was placed onto the prepared dentine surface. The mold was bulk-filled with Fuji II LC® (GC), which was then light-cured for 30 seconds with a Demetron LC curing light (Kerr Corporation, Orange, CA, USA) with a minimum output of 600 mW/cm². After light-curing, the mold was removed and the excess cement, if present, was gently removed from around the base of the RMGIC cylinder with a scalpel. The samples were stored in 37°C water until tested. The materials, their composition, manufacturers, batch numbers, and application details are presented in Table 1.

Subgroups (n = 15) of each group were tested for SBS after 24 hours, six months, and 12 months of storage using a universal testing machine (LRX, Lloyd Instruments, Fareham, Hants, UK). For testing, each sample was immobilized in a device provided with a sliding blade acting like a guillotine, thus loading the dentin-RMGIC interface in shear. A cross-head speed of 0.5 mm/min was used. The fractured specimens were observed under a binocular microscope (Olympus Europe SZH10, Hamburg, Germany) at 40X magnification and the fractures were classified as adhesive (failure at the interface between dentin and RMGIC), cohesive (failure in RMGIC), or mixed (involving both adhesive and cohesive failures).

The results of SBS were analyzed by two-way analysis of variance for the factors “dentin treatment” (with CHX vs without CHX) and “ageing period,” followed by Tukey post hoc pairwise comparison tests. A chi-square test was used for the analysis of the modes of failure. Statistical significance for all tests was set at p < 0.05. Statistical calculations were performed using StatView® Version 5.0 software for Windows (SAS® Institute Inc, Cary, NC, USA).

### RESULTS

The results of SBS along with the results of the statistical analysis are summarized in Table 2. The statistical analysis revealed that SBS was significantly influenced by CHX application and time. At 24 hours, the SBS was not significantly different with or without the application of CHX. After six months and 12 months, however, significant differences were identified: in the subgroups without CHX, SBS increased significantly, as compared to...
24 hours, and it was higher than in the subgroups with CHX; within the CHX subgroups, SBS after six months and 12 months was not significantly different from that after 24 hours.

The results of the failure mode along with the results of the statistical analysis are summarized in Table 3. Fracture mode analysis did not demonstrate any statistically significant differences between or within the groups at any time point.

**DISCUSSION**

In the first part of this *in vitro* work we investigated whether the use of 0.05% CHX interfered with the SBS of a RMGIC to dentin after 24 hours of storage (early/short term) in water at 37°C. Under the conditions of the study, the magnitude of early SBS values without CHX (8.3 MPa) was close to those obtained in the literature with similar adhesion tests and with adhesive failures being predominant. The presence of CHX did not affect the early SBS (9.0 MPa). This finding has also been observed with etch-and-rinse and self-etch adhesives as well as with self-adhesive resin cements used for direct or indirect adhesive restorations. However, adverse effects of CHX on early bond strength have also been reported, and they were attributed to the water content of the 0.05% CHX solution. The authors speculated that after the application of this solution, residual moisture might diminish the adhesive properties of some adhesive systems. In the current study, the air-drying of the dentin surface after CHX application and the hydrophilicity of the RMGIC may explain why such adverse effects were not observed.

In the second part of this *in vitro* work we investigated whether the use of 0.05% CHX interfered with the long-term (six months and 12 months of storage in water at 37°C) SBS of a RMGIC. In the samples with no CHX, after six months a significant increase in SBS, from 8.3 MPa to 12.7 MPa, was determined. After 12 months, SBS was maintained at 12.7 MPa, with predominant adhesive failures as well. These results confirm those of previous studies that observed an increase in bond strength with time, concomitant with the RMGIC maturation. The results are also consistent with studies that have indicated that the cross-link density in RMGIC increases as a result of the slow diffusion of calcium and aluminum ions and that this phenomenon is responsible for the increase in compressive strength with ageing.

Mount described an ion-enriched layer, formed by the displacement of calcium and phosphate ions from apatite by the carboxyl group of glass ionomer liquid, and re-precipitation of ions at the cement-tooth interface with the setting of glass ionomer material. Mitra and others also reported an amorphous zone, which resembles a hybrid layer and is thought to be a reaction product of the RMGIC with inorganic material from dentin, described as a diffusion-based adhesion. Moreover, it may be noted that RMGICs involve a chemical bonding by ionic
interactions of the carboxyl groups of the polyalkenoic acid with calcium ions of hydroxyapatite attached to collagen fibrils. It has been shown that this chemical bonding contributes to the excellent long-term adhesion, microleakage resistance, and dentin sealing ability of these materials. Furthermore, the combination of chemical adhesion and micro-mechanical retention may be beneficial to the long-term resistance to hydrolytic degradation of this hybrid layer.

The CHX-treated group, however, showed no increase in SBS after six months or 12 months of ageing. The results obtained indicate that the presence of CHX impeded the increase in SBS observed in the non-CHX group, and, therefore, the null hypothesis was rejected. This phenomenon has not been reported before, and it may be explained as follows: CHX, which has strong cationic properties, could react with the anionic carboxyl groups of the RMGIC, thus impeding the formation of carboxyl-calcium linkages and therefore reducing the dentin bonding capability of RMGIC. The interference of CHX with the chemical adhesion mechanism of RMGIC could decrease the resistance to hydrolytic degradation of the hybrid layer. Furthermore, CHX may also interfere with the second step of the setting reaction of RMGIC by competing with aluminum ions for the carboxyl groups, thus perturbing the maturation reaction of RMGIC.

The long-term negative effect of CHX on the SBS of the RMGIC investigated does not follow the results obtained by numerous studies for resin composite and etch-and-rinse adhesive systems, which showed that CHX has the potential to minimize the reductions in the resin-dentin bond strengths commonly observed after long-term water storage.

Finally, the current results are in line with the findings of a study that reported a decrease in the physical properties of RMGIC combined with CHX. Future studies with alternative MMP inhibitors that cannot interfere with the bonding or maturation mechanisms of RMGIC should be conducted to investigate if their presence can affect the long-term performance of RMGIC.

CONCLUSIONS
Under the conditions of this in vitro study, the results have shown that the 24-hour SBS of the tested RMGIC to dentin was not affected by CHX; at six and 12 months, however, the CHX-treated samples exhibited lower SBS, which might have been caused by an interference of CHX with both the bonding mechanism and the maturation reaction of the cement.

Finally, the present findings should be interpreted with caution, as the results were obtained under laboratory conditions, and it remains to be seen whether or not CHX could inhibit the in vivo degradation of the hybrid layer at the dentine-RMGIC interface by endogenous MMPs.

Conflict of Interest
The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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REFERENCES


of dentin bonding systems Quintessence International 35(1) 56-60.


