



Year: 2013

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Abstract: **OBJECTIVE:** To investigate the influence of caries infiltrant preconditioning on the shear bond strength of orthodontic resin cements on sound and demineralized enamel. **MATERIALS AND METHODS:** Stainless-steel brackets were bonded to sound or artificially demineralized (14 d, acidic buffer, pH 5.0) bovine enamel specimens using a resin cement or a combination of caries infiltrant preconditioning (Icon, DMG) and the respective resin cement (light-curing composite: Heliolit Orthodontic, Transbond XT, using either Transbond XT Primer or Transbond Plus Self Etching Primer; light-curing resin-modified glass ionomer cement: Fuji Ortho; or self-curing composite: Concise Orthodontic Bonding System). Each group consisted of 15 specimens. Shear bond strength was evaluated after thermo-cycling (10,000×, 5°C to 55°C) at a crosshead speed of 1 mm/min, and data were statistically analyzed by analysis of variance, Mann-Whitney test, and Weibull statistics. Adhesive Remnant Index (ARI) scores and enamel fractures were determined at 25× magnification and were statistically analyzed by regression analyses ($P < .05$). **RESULTS:** The caries infiltrant system significantly increased the shear bond strength of Transbond XT Primer, Transbond Plus Self Etching Primer, and Fuji Ortho in sound specimens, and of all resin cements except for the Concise Orthodontic Bonding System in demineralized enamel. Overall, caries infiltrant preconditioning decreased significantly the number of enamel fractures, but it did not affect ARI scores. **CONCLUSION:** Preconditioning of sound and demineralized enamel with the caries infiltrant system did not impair but rather increased the shear bond strength of most orthodontic resin cements while decreasing the risk of enamel fracture at debonding.

DOI: <https://doi.org/10.2319/052112-409.1>

Posted at the Zurich Open Repository and Archive, University of Zurich

ZORA URL: <https://doi.org/10.5167/uzh-86769>

Journal Article

Accepted Version

Originally published at:

Naidu, Ewelina; Stawarczyk, Bogna; Tawakoli, Pune Nina; Attin, Rengin; Attin, Thomas; Wiegand, Annette (2013). Shear bond strength of orthodontic resins after caries infiltrant preconditioning. *Angle Orthodontist*, 83(2):306-312.

DOI: <https://doi.org/10.2319/052112-409.1>

Shear bond strength of orthodontic resins after caries infiltrant preconditioning

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Submitted: May 2012

NO Color Figures

ABSTRACT

Objectives: To investigate the influence of caries infiltrant preconditioning on the shear bond strength of orthodontic resin cements on sound and demineralized enamel.

Materials and Methods: Stainless steel brackets were bonded to sound or artificially demineralized (14 d, acidic buffer, pH 5.0) bovine enamel specimens using a resin cement or a combination of caries infiltrant preconditioning (Icon, DMG) and the respective resin cement (light-curing composite: Heliolit Orthodontic, Transbond XT, using either Transbond XT Primer or Transbond Plus Self Etching Primer; light curing resin-modified glass ionomer cement: Fuji Ortho; or self-curing composite: Concise Orthodontic Bonding System). Each group consisted of $n = 15$ specimens. Shear bond strength was evaluated after thermo-cycling (10.000x, 5° to 55°C) at a crosshead speed of 1 mm/min and statistically analysed by analyses of variance, Mann-Whitney tests and Weibull statistics. Adhesive remnants index (ARI) scores and enamel fractures were determined at 25× magnification and statistically analysed by regression analyses ($P < .05$).

Results: The caries infiltrant system increased significantly the shear bond strength of Transbond XT Primer, Transbond Plus Self Etching Primer and Fuji Ortho in sound specimens and of all resin cements except Concise Orthodontic Bonding System in demineralized enamel. Overall, caries infiltrant preconditioning decreased significantly the number of enamel fractures, but did not affect ARI scores.

Conclusion: Preconditioning of sound and demineralized enamel with the caries infiltrant system did not impair, but even increased shear bond strength of most orthodontic resin cements, while decreasing the risk of enamel fracture at debonding.

Key words: Enamel; Orthodontic resin; Shear bond strength; Caries infiltrant

INTRODUCTION

The fixation of orthodontic brackets enhances plaque retention and, thus, favors the development of demineralization and initial caries around the brackets when the oral hygiene of the patient is poor. Different attempts were made to increase the caries resistance of enamel prior and during fixed orthodontic treatment, e.g. application of fluoride or casein-phosphopeptide-amorphous calcium phosphate.¹⁻⁴ Schmidlin et al.⁵ suggested the use of an adhesive patch as an intermediate layer under metal brackets to prevent demineralization under and around the bracket and to relocate the bracket-patch margins to areas easier to access for oral hygiene. Also, the use of different (fluoridated) bonding agents and sealants was discussed to protect surrounding enamel from demineralization.^{4;6;7}

However, enamel white spot lesions not only develop during orthodontic treatment, but might be present even at the start of the orthodontic treatment. Tufekci et al.⁸ and Gorelick et al.⁹ found 11% and 24% patients, respectively, with existing white spot lesions at the time of bracket fixation. Thus, preventive strategies in orthodontics also include means to remineralize previously demineralized enamel to allow for bracket fixation.¹⁰

In recent years, low-viscosity resins with a high capability for penetrating initial caries lesions were increasingly studied. Clinical trials showed that the caries progression of infiltrated lesions is significantly reduced.^{11;12} Although originally developed for penetration into carious lesions and occlusion of diffusion pathways, the resin infiltrant also prevented enamel surface demineralization to some extent.¹³ Moreover, existing white spot lesions might be covered and provided with increased stability,^{10;14} thus bond strength of brackets might be increased compared to demineralized enamel without pretreatment.¹⁰

Due to the sealing effect on sound enamel and the stabilization of demineralized enamel, it is conceivable that the caries infiltration technique is beneficial as pretreatment prior to bracket fixation. Therefore, the purpose of this study was to investigate the influence of caries infiltrant

preconditioning on the shear bond strength of orthodontic resin cements on sound and demineralized enamel. The null hypothesis was that preconditioning with the caries infiltration system does not reduce shear bond strength of orthodontic cements.

MATERIALS AND METHODS

Specimen preparation

Three-hundred cylindrical enamel specimens (6.6 mm in diameter) were prepared from the labial surface of freshly extracted bovine crowns, embedded in chemically cured acrylic resin (ScandiQuick, ScanDia, Hagen, Germany) and ground flat with P 180 SiC paper (Struers Willich, Germany). The surfaces were polished under water cooling with 500 SiC paper (Struers, Willich, Germany).

Half of the specimens were demineralized in an acidic buffer (pH 5.0, 37° C,¹⁵) for 14 days to create artificial caries-like lesions. The buffer solution was renewed every second day to keep the pH constant. Sound and demineralized specimens were randomly allocated into 10 groups each (n = 15).

Bonding procedure and application of brackets

Stainless steel brackets for central lower incisors (Discovery, slot 0.56 × 0.76 mm/22 × 30, 8.71 mm² surface area bracket base, Dentaurum, Ispringen, Germany) were bonded to the sound or demineralized specimens, using either one of the resin cements or a combination of caries infiltrant (Icon, DMG, Germany) preconditioning and the respective resin cement. The resin cements comprised two light-curing composites (Heliosit Orthodontic, Transbond XT), one light-curing resin-modified glass-ionomer cement (Fuji Ortho) and one self-curing composite (Concise Orthodontic Bonding System). The resin cement Transbond XT was used either after conventional etching and application of Transbond XT primer or after applying a self-etching primer (Transbond Plus Self Etching Primer). The compositions of the materials are presented in Table 1.

Preconditioning with the caries infiltrant included hydrochloric acid etching (15 % HCl, 2 min, Icon Etch, DMG, Hamburg, Germany), water rinsing (30 s), surface drying by ethanol (30 s, Icon Dry, DMG, Hamburg, Germany) and application of a low-viscosity resin infiltrant (Icon Infiltrant, DMG, Hamburg, Germany). The resin infiltrant was applied twice (3 min and 1 min), each application followed by light-curing (800 W/cm², bluephase, IvoclarVivadent, Schaan, Liechtenstein) for 40 s at 2 mm distance to the surface. In half of the sound or demineralized enamel groups, respectively, specimens were preconditioned with the caries infiltrant system prior to resin cement application, while in the remaining groups the brackets were bonded using the resin cements accordingly to the manufacturers' recommendations (Table 2).

The brackets were fixed on the specimens with a standardized load of 500 g. Excess material was carefully removed with microbrushes at 2.5× magnification. Light-curing of the cements was performed accordingly to the manufacturers recommendations at a distance of 5 mm. The self-curing system Concise Orthodontic Bonding System was allowed to set for 10 min.

Shear bond strength measurement

Prior to shear bond strength testing, the specimens were thermo-cycled 10.000 times between 5° and 55°C (Willytec, Gräfelfing, Germany, dwell time: 20 s in each bath, transfer time: 10 s).

Shear bond test was performed with an universal testing machine (Z010, Zwick, Ulm, Germany). A shear force was applied through a chisel-shaped loading device at a crosshead speed of 1 mm/min parallel to the specimens surface. Load at fracture was recorded and shear bond strength was calculated according to the following equation: $S = F/A$, where S is shear bond strength, F is load at failure (N), and A represents the adhesive area (mm²) (TestXpert 11.02, Zwick, Ulm, Germany).

The debonded area was examined with a stereomicroscope at 25x magnification (M3B, Wild, Heerbrugg, Switzerland) for assessment of the Adhesive Remnant Index (ARI) and enamel fractures.¹⁶ ARI scores ranged from 0 to 3: 0 = no adhesive was left on the enamel surface, 1 =

less than half the adhesive was left on the enamel, 2 = half or more of the adhesive was left on the enamel, 3 = all of the adhesive was left on the enamel.

Statistical analysis

Mean shear bond strength (\pm standard deviation) for each group was computed. Kolmogorov-Smirnov and Shapiro-Wilk tests were applied to check the assumption of normality. Although data were not normally distributed in two of twenty groups, data were analysed by three-way ANOVA, factors being the substrate (sound/demineralized enamel), the pretreatment mode and the kind of primer/resin cement to check for possible interactions. As all factors and interaction were found to be significant, two-way ANOVAs were further applied within sound and demineralized enamel groups, factors being the pretreatment mode and the kind of primer/resin cement.

Mann-Whitney tests were applied to analyse differences between specimens with and without caries infiltrant preconditioning (separately for the primers/resin cements and the substrate). Differences between the primers/resin cements were analysed by Kruskal-Wallis tests (separately for sound and demineralized specimens and specimens with and without caries infiltrant preconditioning) followed by pairwise Mann-Whitney tests and Bonferroni correction. Differences between shear bond strength values of sound and demineralized enamel groups were also analysed by Mann-Whitney tests.

Weibull statistics (Weibull modulus m , characteristic bond strength σ_0) was calculated using the maximum likelihood estimation method at 95% confidence level (MINITAB, State College, PA, USA).

To analyse the effect of the caries infiltrant preconditioning on ARI scores and enamel fractures, data were statistically analysed by ordinal or linear regression analysis, respectively. In all tests, the level of significance was set at $P < .05$.

RESULTS

Shear bond strength values of sound and demineralized enamel groups are presented in Figure 1. Three- and two-way ANOVAs showed all factors and all interactions to be significant with respect to shear bond strength ($p > 0.004$).

Preconditioning with the caries infiltrant system increased the shear bond strength of Transbond XT Primer ($p = 0.009$), Transbond Plus Self Etching Primer ($p = 0.001$) and Fuji Ortho ($p = 0.011$) in sound specimens and of all resin cements ($p > 0.009$) except Concise Orthodontic Bonding ($p = 0.265$) System in demineralized enamel. Generally, lowest shear bond strength values were observed for Fuji Ortho, independently of the preconditioning and the substrate (sound or demineralized enamel). Without caries infiltrant preconditioning, shear bond strength of Heliosit Orthodontic ($p > 0.001$) and Fuji Ortho ($p > 0.001$) was significantly reduced in demineralized compared to sound enamel. All other comparisons between sound and demineralized enamel were not significantly different.

The Weibull parameters are presented in Table 3. In most groups, characteristic strength (σ_0 values) increased when specimens were preconditioned with the caries infiltrant system. Weibull moduli m were remarkably low in groups where brackets were bonded with Fuji Ortho and when Heliosit Orthodontic and Transbond XT Primer were used on demineralized enamel without Icon preconditioning.

ARI scores and enamel fractures are presented in Table 4. Regression analyses revealed that preconditioning with the caries infiltrant system was significant with respect to enamel fractures but not with respect to the ARI scores. Enamel fractures occurred only in demineralized enamel, but were significantly reduced when specimens were preconditioned with Icon. ARI scores with

and without caries infiltrant preconditioning were not significantly different within each primer/resin cement.

DISCUSSION

To the first time, a caries infiltrant system was used for preconditioning of sound and artificially demineralized enamel prior to bracket fixation. The null hypothesis cannot be rejected as Icon preconditioning did not reduce, but even increased shear bond strength of some of the resin cements.

The results of the present study are promising, as the preconditioning with the caries infiltrant system enhanced shear bond strength in most groups and reduced the frequency of enamel fractures in demineralized enamel at bracket debonding. In the light of previous studies showing that the caries infiltrant system reduced progression of initial caries lesions and protected both sound and demineralized enamel from acid dissolution, this suggests that the range of application of the caries infiltrant system might be extended to the preconditioning of enamel prior to fixed orthodontic treatment.

Shear bond strength was tested on bovine enamel which has been proposed as suitable alternative for human enamel in shear bond strength testing, although slightly different values between human and bovine enamel were shown.¹⁷ Artificial carious lesions were created according to previous studies^{18;19} and exhibit the typical histological structure of initial enamel caries (intact surface layer, lesion body, demineralisation front).²⁰ Moreover, they have the advantage of a higher homogeneity, allowing for a better standardization than natural lesions in human teeth. Preconditioning with the caries infiltrant system followed the recommendations of the manufacturer, thus enamel was etched with hydrochloric acid for 120 s. As the surface layer of artificial caries lesions is slightly thinner as compared to natural lesions, it might be assumed that extensive etching with hydrochloric acid does not only remove the surface layer but also induce a breakdown of the lesion. However, in fact it was shown that hydrochloric acid etching for

120 s does remove the surface layer of artificial lesions, without destroying the underlying structure.¹⁴ Complete removal of the surface layer was shown to be essential for the penetration of the TEGDMA-based resin infiltrant.²¹

Preconditioning with the caries infiltrant system increased shear bond strength of the resin cements on artificial enamel lesions most likely due to a deeper penetration of the resin infiltrant into the body of the lesion compared to the primer or paste, respectively, of the orthodontic cements. The frequency of fractures in demineralized enamel was significantly reduced compared to specimens without Icon preconditioning, indicating a stabilizing effect of the resin infiltrant.

Monomer formulations with an increased TEGDMA content have a high penetration capability²² and were shown to induce the formation of a thick oxygen-inhibited layer,²³ which probably allows a chemical connection of the resin infiltrant to the monomers of the primer or paste. Conventional adhesives are able to penetrate carious lesions to some extent, thus it is assumed that the primers of the tested systems also might penetrate demineralized enamel partly and strengthen the outermost part of the infiltrated enamel when applied after Icon preconditioning. However, Concise was shown to exhibit a relatively high penetration coefficient,²⁴ which might explain why this material did not benefit from the preconditioning with the caries infiltrant system.

From the results of this study it becomes evident that especially resin cements without an additional primer (Heliosit Orthodontic and Fuji Ortho) benefit from the preconditioning with the caries infiltration system, as their penetration capability is limited²⁴ probably due to the high amount of Bis-GMA and UDMA²² (Heliosit Orthodontic) and the high filler content (Fuji Ortho),²⁵ respectively. In case of Fuji Ortho, penetration into demineralized enamel is further hampered as etching with polyacrylic acid less efficient compared to phosphoric acid,²⁶ probably resulting in an incomplete removal of the intact surface layer. Also, acidic monomers of self-etching adhesives

were shown to be unable to erode the surface layer of artificial enamel lesions adequately to allow for penetration of the adhesive.²⁷

Therefore, the penetration of Transbond Plus Self Etching Primer might be distinctly increased by etching with hydrochloric acid during Icon preconditioning, thus resulting in significantly higher shear bond strength values. Even on sound enamel, preconditioning with the caries infiltrant system had a slightly positive effect on shear bond strength, probably due to the high wettability of the TEGDMA-resin.²²

The results were confirmed by the Weibull statistics, which mostly showed higher characteristic strength σ_0 values when specimens were preconditioned with the caries infiltrant system. As lower Weibull moduli indicate larger variation in measured strengths and suggest poorer reliability of a material, the shear bond strength data in specimens bonded with Fuji Ortho and in demineralized enamel bonded with Heliostat Orthodontic and Transbond XT Primer without Icon preconditioning should be interpreted with caution.

Overall, bond strength was not impaired but even enhanced by caries infiltrant preconditioning, confirming the results of previous studies which showed that caries infiltrant application increased shear bond strength values of etch & rinse and self-etching adhesives.^{18;19} From a clinical point of view it is of relevance that the frequency of enamel fractures at debonding was decreased by the preconditioning, while in the same time the ARI scores were not significantly changed, indicating that the amount of residual resin to be removed at debonding is not affected by this technique.

CONCLUSIONS

- The preconditioning with the caries infiltrant system increased shear bond strength of most orthodontic resins to sound and demineralized enamel, while at the same time the risk of enamel fractures at debonding was reduced and the amount of residual resin remained unchanged.

ACKNOWLEDGMENT

The study was supported by DMG, Germany.

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Figure 1. Shear bond strength (mean \pm standard deviation, MPa) of sound (a) and demineralized (b) enamel. Within each primer/resin cement, significant differences between enamel with and without caries infiltrant preconditioning are marked by an asterisk. Significant differences between the primers/resin cements are marked with different small letters within sound and demineralized enamel separately for groups with and without caries infiltrant preconditioning.

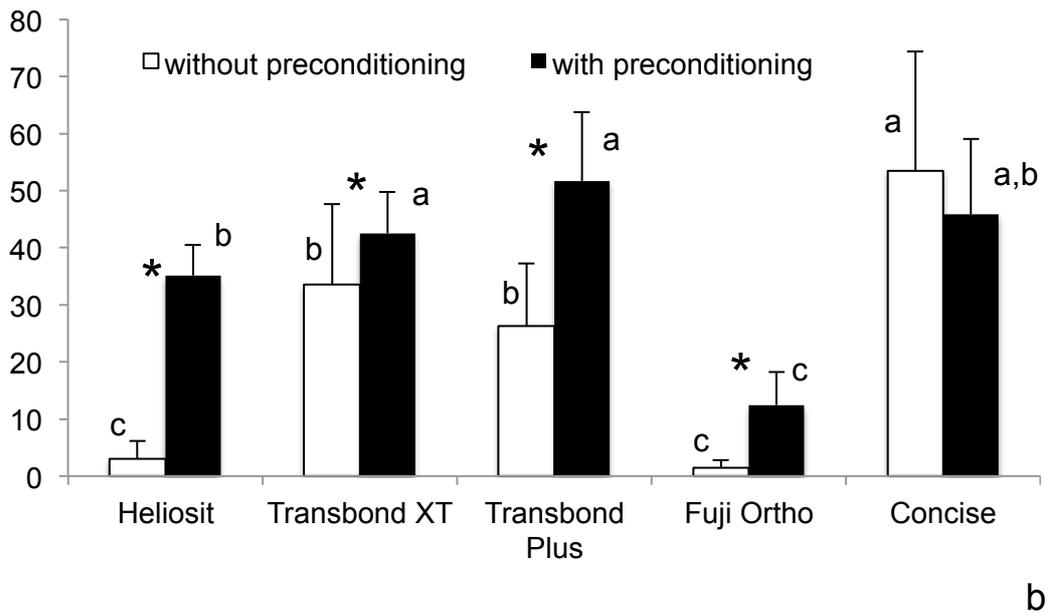
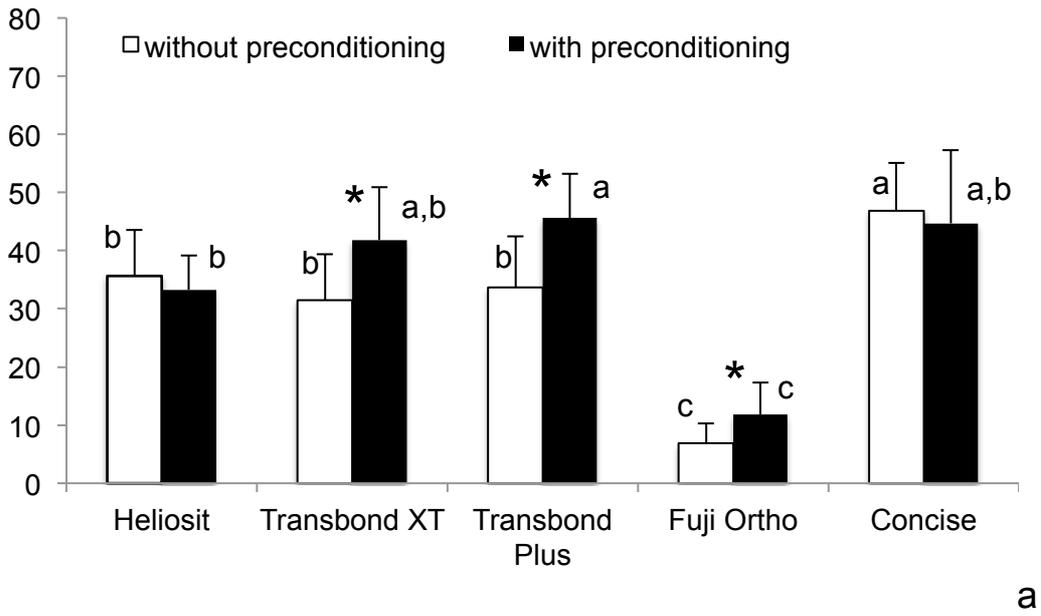


Figure 1