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Effects of chlorhexidine-containing adhesives on the durability of resin–dentine interfaces

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ABSTRACT

Objectives: This study evaluated the effect of addition of diacetate CHX in different concentrations into two simplified etch-and-rinse (ER) adhesive systems (XP Bond [XP] and Ambar {AM}) on the ultimate tensile strength (UTS), degree of conversion (DC), 60-day cumulative water sorption (WS), solubility (SO) and CHX release (CR) as well as the immediate (IM) and 1-year (1Y) resin–dentine bond strength (μTBS) and nanoleakage (NL).
Methods: Ten experimental adhesive systems were formulated according to the addition of CHX diacetate (0 [control], 0.01, 0.05, 0.1 and 0.2%) in the two ER. For UTS and DC, specimens were constructed and tested after 24 h. For WS, SO and CR, after specimens build-up, they were stored in water and the properties measured after 60 days. The occlusal enamel of fifty molars was removed and the adhesives were applied in dentine surface after 37% phosphoric acid etching. After composite resin build-ups, specimens were longitudinally sectioned to obtain resin–dentine bonded sticks (0.8 mm^2). Specimens were tested in tension at 0.5 mm/min in the IM or 1Y. For NL, 2 bonded sticks from each tooth were prepared and analyzed under SEM. The data were submitted to appropriate statistical analysis ($\alpha = 0.05$).
Results: The addition of CHX did not influence UTS, DC, WS and SO ($p < 0.05$). Higher CR was observed in adhesives with higher concentration of CHX ($p < 0.05$). After 1Y, significant reductions of μTBS and increases of NL were observed in the control groups ($p < 0.05$). Reductions of μTBS and increase of NL over time were not observed (AM) for CHX-containing adhesives or it was less pronounced than the control (XP) regardless of the CHX concentration.
Conclusions: The addition of CHX diacetate in concentrations until 0.2% in the simplified ER adhesive systems may be an alternative to increase the long-term stability of resin–dentine interfaces, without jeopardizing the adhesives' mechanical properties evaluated.

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1. Introduction

The most important concern regarding contemporary adhesive restorations is their limited durability in vivo,[1,2] which results in millions of dental care dollars spent annually on replacement of these restorations.[3] Clinically, the most frequently occurrence of bonding failure is the retention loss of adhesive restorations as well as marginal defects, such as lack of marginal adaptation and marginal discoloration.[1,2]

These clinical concerns are mainly observed with simplified self-etch and etch-and-rinse (ER) adhesive systems[1,2] as they are more prone to water sorption[4] and therefore susceptible to the swelling effect of water on the polymer produced after light-curing. However, not only the polymer is susceptible to degradation. Host-derived matrix metalloproteinases [MMPs][5] and cysteine catepsines,[6] activated after adhesive application[7–9] has been claimed to degrade the collagen fibrils.

Several approaches to inhibit the activities of these host-derived enzymes were proposed in an aim to prolong the clinical lifetime of bonding interfaces.[8,9] It was demonstrated that collagen fibrils could be preserved after storage in a solution containing chlorhexidine [CHX], a non-specific synthetic protease inhibitor.[5] This was further confirmed by in vivo studies in deciduous[10] and permanent teeth.[11] These studies did not observe degradation of the collagen fibrils after application of an aqueous solution of 2% CHX digluconate on acid-etched dentine, before adhesive application.[10,11]

In spite of the beneficial findings of the use of 2% CHX for 60 s, as a non-rinse primer on etched dentine, this procedure adds an extra step to the bonding protocol, which is against the clinicians' preference for simplification. In face of that other studies evaluated the impact of CHX incorporation in the acid conditioner[12,13] or in the adhesive solution.[14–17] The CHX inclusion in the phosphoric acid was capable to preserve the resin–dentine degradation after 6 and 24 months of water storage.[12,13] When it comes to the inclusion of CHX or other proteases inhibitors in primers and/or adhesives, controversial results have been published.[14–19] Variations in the amount of CHX concentration and the type of bonding strategy evaluated could be responsible for such variability.[14–17]

So far, only one study has evaluated the impact of CHX addition in a simplified ER adhesive.[17] The authors did not observe preservation of the resin–dentine microtensile bond strength (μTBS), which was attributed to the low concentration of CHX (0.05%) added to the bonding solution. Previous studies demonstrated that very low CHX concentrations ranging from 0.2 to 0.002%[20,21] could prevent dentine bonding degradation; however in these studies CHX was used as a primer on etched-dentine. The incorporation of CHX into the bonding solution likely restricts the availability of this protease inhibitor to the bonded environment by polymer entrapment.

Thus, the optimal concentration of CHX that may be added in the adhesive to produce stable bonds without jeopardizing other mechanical properties of the adhesive layer is yet to be addressed. Therefore, this in vitro study was designed to evaluate the effect of diacetate CHX concentration into two simplified ER adhesive systems on the ultimate tensile strength, degree of conversion, 60-day cumulative water sorption and solubility of the adhesives as well as the immediate and 1-year resin–dentine μTBS and nanoleakage. The following null hypotheses were tested: (1) the different CHX concentrations will not result in differences in the mechanical properties of the two adhesive tested and; (2) the different CHX concentrations will not result in differences in the immediate and 1-year μTBS and nanoleakage of the adhesives.

2. Methods and materials

2.1. Formulation of the experimental adhesives

The experimental adhesives used in the present study were formulated by using the simplified ER adhesive systems XP Bond [XP] (Dentsply, York, PA, USA) and Ambar [AM], (FGM, Joinville, SC, Brazil). Detailed compositions, mode of application and batch number of the adhesives are depicted in Table 1.

Ten experimental adhesive systems were formulated according to the addition of different concentrations of CHX diacetate (99.9% pure, Sigma Chemical, St. Louis, MO, USA) (wt%): 0 (control), 0.01, 0.05, 0.1 and 0.2%. The CHX was added to the bonding resin and mechanically mixed by a motorized mixer (stirring).

2.2. Teeth preparation and bonding procedures

Fifty caries-free extracted human third molars, collected from patients with ages from 18 to 35 years old, were used. The teeth were collected after the patient's informed consent. The University Ethics Committee approved this study under protocol number 1693/09. Teeth were disinfected in 0.5% chloramine, stored in distilled water and used within 3 months after extraction. A flat dentine surface was exposed on each tooth after wet grinding the occlusal enamel with 180-grit SiC paper. The enamel-free, exposed dentine surfaces were further polished with # 600-grit silicon-carbide paper for 60 s to standardize the smear layer.

The adhesives were applied as per manufacturers' instructions (Table 1) and they were light cured with a LED light for 10 s at 600 W/cm^2 (Radii-cal, SDI, Bayswater, Victoria, Australia). Resin composite blocks (Opallis, FGM) were build-up on the bonded surfaces (3 increments of 1.0 mm each) and were individually light activated for 40 s. A single operator in an environment with controlled temperature and humidity carried out all bonding procedures. Five teeth were used for each experimental group.

After storage of the bonded teeth in distilled water at 37 °C for 24 h, they were longitudinally sectioned in both "x" and "y" directions across the bonded interface with a diamond saw in a Labcut 1010 machine (Extec Corp., Enfield, CT, USA), under water cooling at 300 rpm to obtain bonded sticks with a cross-sectional area of approximately 0.8 mm^2. The number of premature failures (PF) per tooth during specimen preparation was recorded. The cross-sectional area of each stick was measured with the digital calliper to the nearest 0.01 mm and recorded for subsequent calculation of the μTBS (Absolute