



Long-term effect of chlorhexidine on the dentin microtensile bond strength of conventional and self-adhesive resin cements: A two-year in vitro study



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ABSTRACT

Since degradation of the adhesive interface plays an important role on dental restoration failure overtime, bonding protocols containing metalloproteinase synthetic inhibitors could be a valuable approach to preserve the bond strength of indirect restorations. A flat dentin surface was created on 40 recently extracted non-carious human third molars ($n=10$). Resin-composite blocks were randomly cemented using two resin cements: a self-adhesive and a conventional. A buffer-free 2% chlorhexidine digluconate solution was used as dentin pretreatment on experimental groups. Microtensile bond strength test was performed immediately and after specimen aging for two-years in artificial saliva. Fracture patterns were determined by SEM. Even after significant reduction in bonding effectiveness with aging, dentin bond strength values of the conventional resin cement remained higher compared to those of the self-adhesive resin cement especially when chlorhexidine pretreatment was performed. No statistical differences were observed between immediate and aged specimens luted with the self-adhesive resin cement. Chlorhexidine was effective to preserve dentin bond strength of indirect restorations when the conventional resin cement was used.

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1. Introduction

Resin cements are routinely used in dentistry for luting composite crowns, all ceramic restorations, and posts. A stable union between resin bonding materials and the tooth substrate is highly important to determine the durability of dental restorations [1]. Since the clinical success of indirect restorative procedures depend in part on the technique and materials used for luting [2,3], different bonding mechanisms, methods to preserve the adhesive interface should be further exploited. Regarding the bonding mechanisms of different cements, interest has been

increasingly focused on the use of self-adhesive resin cements for: (i) no dental substrate pretreatment is required; (ii) chemical adhesion to dental substrates is promoted [4]; and (iii) relative moisture resistance adhesive interfaces are created [5].

Despite significant improvements, the adhesive interface remains the weakest link of restorations especially when dentin is involved. Two major mechanisms are involved in the loss of dentin bond strength over time: (i) hydrolytic degradation of hydrophilic resin within the hybrid layer [7] and (ii) deterioration of the dentin collagen fibrils [7,8]. Self-adhesive cements differ from conventional or self-etch resin cements for their interaction with dentin is only superficial due to limited decalcification, low diffusion, and partial-exposure of collagen fibrils at the base of the adhesive interface [9]. Meanwhile, the use of conventional resin cements creates a discrepancy between the etching depth and resin penetration into dentin. As a consequence, a zone of exposed collagen non-infiltrated by resin monomers at the base of hybrid layer is formed [10,11]. Deterioration of such unprotected collagen fibrils plays an important role on the degradation of the adhesive interface [12,13] resulting in loss of bond strength [14] and

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consecutively reducing adhesive restorations durability [8]. Host-derived enzymes such as metalloproteinases (MMP) have an important role on degradation of resin–dentin adhesive interfaces [12–14]. MMP are a group of mammalian zinc-and-calcium dependent enzymes capable to hydrolyze collagen fibrils [15,16]. Human dentin contains at least MMP –2, –3, –8, –9, –13, –20 [15–18]. Some of these gelatinolytic/collagenolytic enzymes can be expressed by odontoblasts [19] and are activated from their latent form [20] when pH along the dentin substrate drops [21].

Similarly to adhesive procedures involving self-etching adhesives, increase in MMP expression by the dentin–pulp complex [19] and increased collagenolytic activity [22] to near-maximum levels [20] might be expected when self-adhesive cements are bonded to dentin. In addition, residual unpolymerized acidic monomers might continue to etch the dentin substrate [23] possibly contributing to MMP activation. These events may contribute to resin–dentin bond degradation over time [19,20,22] when low pH self-adhesive resin cements are bonded to dentin. In this context, chlorhexidine, which also presents antibacterial properties [24], has been studied as a promisor synthetic MMP inhibitor at low concentrations [25]. Dentin pretreatment with chlorhexidine has been proven to reduce loss of bond strength over time when total-etch systems are used [14,26,27]. Chlorhexidine potent inhibitory effect against endogenous MMP in dentin is relevant for the current adhesive prosthetic dentistry procedures on account of the degradation of resin–dentin bonds that occurs over time [17,28,29]. Since conflicting information can be found on literature regarding the use of MMP synthetic inhibitors to preserve dentin bond strength of resin cements, the aim of this study was to investigate the long-term bond strength of indirect restorations luted with different resin cements to chlorhexidine-pretreated dentin. The tested hypotheses were that: (i) conventional resin cements produce higher dentin bond strengths when compared to self-adhesive cements irrespective of aging or dentin pretreatment with chlorhexidine; (ii) dentin pretreatment with chlorhexidine reduces loss of bond strength after aging when a self-adhesive resin cement is used; and (iii) dentin pretreatment with CHX reduces resin–dentin bond strength loss of conventional resin cements after aging.

2. Material and methods

2.1. Tooth preparation

Forty recently extracted non-carious third molars were obtained after patient informed consent under a protocol analyzed and approved by the Ethical Committee of the Piracicaba Dental School, University of Campinas, SP, Brazil. Teeth were collected from eighteen to twenty-two year old patients and stored at 4 °C in 0.02% sodium azide solution for up to one month before use. A flat dentin surface perpendicular to the tooth longitudinal axis was obtained by sectioning off occlusal enamel to expose medium

dentine (Isomet 1000 Precision Saw Buehler, Lake Bluff, IL, USA). Dentin roughness was standardized with 600-grit SiC paper (BuehlerMet, Buehler, Lake Bluff, IL, USA) for 1 min under water cooling. Cylindrical composite blocks were prepared using a nanofilled light-activated resin composite (Filtek Supreme Z-350, shade A2, 3M ESPE, St Paul, MN, USA). Three incremental layers measuring no more than 2 mm in thickness each were placed into a Teflon mold (5 mm in thickness and 10 mm in diameter) and individually light-cured using a quartz-tungsten halogen (QTH) unit (3M Curing Light, 3M ESPE) with irradiance of 550 mW/cm². Resin blocks were heat treated at 110 °C for 5 min inside an inlay composite chamber (Fotoceram, Goiânia, GO, Brazil) to improve double bond conversion. One side of the composite block was abraded with 600-grit SiC paper (BuehlerMet, Buehler, Lake Bluff, IL, USA) under water cooling to create a flat surface with standardized roughness. The composite blocks were ultrasonically cleaned in distilled water for 5 min, blow-dried, treated with a prehydrolyzed silane agent (Ceramic Primer, 3M-ESPE) for 1 min and blow-dried before bonding.

2.2. Luting procedures

Two resin cements were used to lute the indirect restorations: one self-adhesive luting cement (RelyX U100, 3M-ESPE) and one conventional dual-cured cement (RelyX ARC, 3M-ESPE) (Table 1). Teeth were randomly assigned to four groups ($n=10$) according to resin cement used (ARC or U100) and dentin pretreatment (distilled water or chlorhexidine): (1) ARC, (2) ARC/CHX, (3) U100 and (4) U100/CHX. Adhesive procedures were carried out in a controlled environment with a temperature of 24 °C and a relative humidity of 60%. For all groups, dentin moisture control was performed with sterilized lint-free absorbent papers before and after dentin pretreatments. The absorbent papers were gently placed on top of the flat dentin surface and replaced after 5 s until visible water was no longer absorbed and a surface with a slight glossy appearance was observed. For group ARC/CHX, the dentin surface was etched with 37% phosphoric acid (Scotchbond Etchant, 3M ESPE) for 15 s and rinsed with water for 30 s; excess moisture was removed with absorbent paper. Chlorhexidine pretreatment was performed and consisted of light-pressure application of a 50 μ L aliquot of a phosphate buffer-free 2% chlorhexidine digluconate solution (Clorhexidina s, FGM, Brazil) (pH 6.8) for 60 s, using a sterilized disposable microbrush. Excess moisture was removed once again with absorbent papers and one coat of primer (Adper Scotchbond Multi-Purpose, 3M ESPE) was applied actively for 10 s. The primer was gently blow-dried followed by active application of one coat of adhesive (Adper Scotchbond Multi-Purpose, 3M ESPE) for 10 s. Light-activation was performed for 10 s. The indirect restoration was then luted with RelyX ARC. For group U100/CHX, moisture control was performed and chlorhexidine was applied on the smear layer-covered dentin for 60 s with a sterilized microbrush. Excess moisture was removed with absorbent paper and the indirect restoration was luted with RelyX

Table 1
Materials, composition and manufactures.

Brand name	Composition	Manufacturer
Scotchbond etchant	37% Phosphoric acid	3M ESPE Dental Products, St Paul, MN, USA
RelyX ARC	TEGDMA, bis-GMA, zirconia/silica filler (67.5 wt%) initiators	3M ESPE Dental Products, St Paul, MN, USA
RelyX U100	Phosphoric acid methacrylates, dimethacrylates, inorganic fillers (72 wt%), fumed silica, initiators	3M ESPE Dental Products, St Paul, MN, USA
Filtek Z-350	bis-GMA, UDMA, TEGDMA, Ethyl methacrylates, inorganic fillers, photoinitiators	3M ESPE Dental Products, St Paul, MN, USA
Adper Scotchbond	Primer: HEMA, polyalkenoic acid methacrylate copolymer. Adhesive: bis-GMA, HEMA, photoinitiators	3M ESPE Dental Products, St Paul, MN, USA
Clorhexidina s	2% Chlorhexidine	FGM, Joinville, SC, Brazil

Abbreviations: Bis-GMA=bisphenol A-glycidyl methylmethacrylate; HEMA=hydroxyethyl methacrylate; UDMA=urethane dimethacrylate; TEGDMA=triethylene glycol dimethacrylate.

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