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Original Research Article

Effect of 2% chlorhexidine treated coronal dentin on bond strength of two self-adhesives cements

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Abstract

Introduction: Despite advances in adhesive dentistry, lasting bonds between indirect restorations and dentin remain a challenge. **Objective:** Therefore, the aim of this study was to evaluate the effect of chlorhexidine on the bond strength of self-adhesive cements to dentin after storage for 24 h and 90 days. **Material and methods:** Forty-eight human third molars crowns were included in polyvinyl chloride matrices using acrylic resin and worn to expose a flat dentin area. The specimens were randomly distributed into 8 groups (n = 12) in accordance with the self-adhesive cement (Rely X U200 and Smart Cem 2), the dentin treatment (application or not of 2 % chlorhexidine digluconate solution for 1 min) and the storage period (24 h or 90 d). Two cylinders' cements (1.4 x 1 mm) were constructed on the dentin surfaces of each sample, stored in distilled water for 24 h or 90 days and submitted to a micro-shear test. Subsequently, a failure analysis was performed to classify the failure into adhesive, cohesive, and mixed. Data were subjected to ANOVA and Tukey (0.05) tests. **Results:** There was a statistically significant difference between the materials (Rely X U200 > Smart Cem 2 – $p < 0.05$) regardless of the surface treatment and the storage period. **Conclusion:** The chlorhexidine application did not affect the immediate or delayed bond strength values, regardless of the self-adhesive cement.

Introduction

Despite advances in adhesive dentistry, lasting bonds between indirect restorations and dentin remain a challenge [7, 8, 14]. Two interfaces are created during the luting process. One is located between the cement and the indirect restoration and another is between the adhesive system and the dental surface [21]. The establishment of integrity and stability within these complex interfaces increases bond strength and fracture resistance of the restoration and the tooth structure [9, 17], while guaranteeing long-term success of the restorative treatment. Deterioration of the hybrid layer can lead to microleakage, resulting in staining, recurrent caries, postoperative sensitivity, or debonding of the restoration [5, 14].

Two major mechanisms are involved in the loss of bond strength: hydrolytic degradation of the hybrid layer and deterioration of collagen fibrils [1-4]. This degradation can be caused by the action of metalloproteinases (MMP), which are proteolytic enzymes existing in dentin that are capable of deteriorating the organic matrix of demineralized dentine [3, 6].

Self-adhesive resin cements can be used to eliminate pretreatment of the tooth structure in order to simplify the adhesive technique and reduce technique sensitivity [16, 18]. However, due to its high viscosity and low etching capacity, the bond strength of self-adhesive resin cements is lower than that of conventional resin cements and adhesive systems for direct resins [10, 11, 16, 18]. In an attempt to optimize the bond strength between resin cements and tooth surfaces, surface treatments with different conditioning agents have been suggested.

Chlorhexidine is widely used as an antibacterial agent and has a broad spectrum of activity against oral bacteria [12, 13]. This solution has an inhibitory effect on the activity of metalloproteinases (MMP) on dentin, which can prevent collagen collapse and the corresponding degradation and disintegration of the bond interface [1-4, 9, 15, 19, 20]. There exist only a few reports on the evaluation of the long-term bonding of self-adhesive cements, especially after the use of antimicrobial agents like chlorhexidine.

The aim of this study was to evaluate the effect of chlorhexidine surface treatments on the immediate (1-day storage) and long-term (90-day storage) micro-shear bond strength between dentin and two self-adhesive resin cements. The hypotheses of this study were: (1) chlorhexidine treatment of the dentin substrate increases bond strength values and (2) the bond strength values are statistically different between the self-adhesive resin cements tested.

Material and methods

This study was submitted and approved by the Institutional Review Board regarding ethical aspects (protocol #132.616).

Specimens of dentin were obtained from caries-free, erupted third molars of patients of both genders, aged between 18 and 25 yr. Forty-eight extracted third molar teeth were selected, cleaned, and stored in a 0.5 % (v./v.) chloramine-T solution for up to 2 months after extraction.

The roots of the teeth were sectioned 1 mm below the cemento-enamel junction. Dentin substrate specimens were prepared by sectioning the crowns with a low speed diamond saw (Isomet 1000; Buehler, Germany) perpendicular to the long axis of the tooth to remove the occlusal enamel and to expose the flat middle-third dentin surface direction.

The tooth sections (enamel or dentin) were then cast in polyvinyl chloride (PVC) rings (1.2 x 2.5 cm^2) using acrylic resin (Jet; Articles Dental Classic Ltd., São Paulo, SP, Brazil) and wet-sanded with 400- and 600-grit silicon carbide paper (Metaserv; Buehler, Germany). Thereafter, they were placed under water in an ultrasonic cleaner for 5 min.

The specimens were then randomly assigned to 8 groups (n = 12) based on the type of surface treatment (i.e. no treatment/control or application of 2 % (v./v.) chlorhexidine digluconate solution), luting agent (Rely X U200 [3M/ESPE, St. Paul, MN, EUA] or Smart Cem 2 [Dentsply, São Paulo, SP, Brazil]), and storage period (1 day or 90 days).

During specimen preparation, exposed surfaces of enamel and dentin were wet-sanded with 600-grit silicon carbide paper for 1 min to standardize the smear layer. Afterward, the samples were subjected to their respective treatments. The control group received no surface treatment apart from the standardization of the smear layer. The chlorhexidine digluconate group was actively treated with a solution of 2% chlorhexidine digluconate for 60 s with the aid of a moistened micro-brush. Subsequently, the specimens were thoroughly washed.

Following the dentin surface treatments, a split silicone mold (diameter: 1.4 mm, height: 1 mm) was clamped to the surfaces and filled with the respective self-adhesive resin cement. The luting agent was light-cured continuously for 40 s at 1,100 mW/cm^2 (Poly Wireless; Kavo, Joinville, SC, Brazil). Ten minutes after light curing, the bonded specimens were freed from their molds and individually stored in distilled water at 37°C for 1 day or 90 days.

Shear bond strength was tested using a universal testing machine (EMIC DL2000; EMIC, São José dos Pinhais, PR, Brazil). The wire-loop method was adopted at a crosshead speed of 0.5 mm/min. Mean and standard deviation values were calculated for each group.

The failure modes were evaluated at x57 magnification under a stereomicroscope (SZX9; Olympus, Tokyo, Japan). Failure was classified as either mainly adhesive, mainly cohesive within the resin cement, cohesive within the enamel or dentin, or a mixture of adhesive and cohesive.

The micro-shear bond strength values were separately subjected to analysis of variance (three-way ANOVA) while considering the factors of surface treatment (no treatment or chlorhexidine digluconate), self-adhesive cement (Rely X U200 or Smart Cem 2), and storage period (1 or 90 d). Additionally, Tukey's test ($\alpha = 0.05$) was performed. The percentage and frequency of the types of failure were subjected to the chi-square test.

Results

The mean values (MPa) and standard deviation (SD) for the different groups after micro-shear bond strength test are shown in table I. According to the analysis of variance, there was a statistically significant difference for only the self-adhesive cement used. The Rely X U200 showed higher bond strength values of micro-shear compared with the Smart Cem 2 regardless of the surface treatment and the storage period. There was no statistical difference for the treatment factor (with or without chlorhexidine application) and storage period (24 h or 90 d).

Regarding the failure mode, there were no significant differences among the groups ($p > 0.05$). The predominant failure mode was adhesive for all groups at 1 day and 90 days. Cohesive and mixed failures were found in some samples of random character.

Table I – Mean (MPa) and standard deviation (SD) for the different groups after micro-shear bond strength test, in enamel (n=12)

Substrate treatment	Material	Period (d)	Mean (±SD)*
No treatment (control)	Rely X U200®	1	10.33 (±4.9)
		90	12.52 (±6.4)
	Smart Cem 2®	1	3.53 (±1.28)
		90	1.08 (±0.61)
Chlorhexidine	Rely X U200®	1	9.33 (±5.4)
		90	14.06 (±4.7)
	Smart Cem 2®	1	2.32 (±0.92)
		90	1.33 (±0.71)

* Means followed by different superscript letter are significantly different ($p<0.05$)

Discussion

In accordance with other studies [12, 14, 19], the substrate treatment with chlorhexidine did not affect bond strength values of self-adhesive cements. As a result, the first hypothesis was rejected. In contrast, other authors [6, 9] indicate that chlorhexidine may negatively affect the bond strength of self-adhesive resin cements. No evidence of this phenomenon was observed in the present study. The results of Stape *et al.* [20] suggest an incompatibility between chlorhexidine and RelyX U100. This reduced bond strength can be explained by the formation of chemical precipitates that act as physical barriers to penetration of the cement as well as differences in the concentration, time of chlorhexidine application, or the type of cement used. Chlorhexidine did not improve or decrease bond strength with the materials tested in the present study. However, it can be considered an effective irrigant that can remove part of the smear layer, open some dentinal tubules, and simplify the irrigation protocol.

Lührs *et al.* [14] and Shafiei and Memarpour [19] verified a decrease in bond strength values of self-adhesive cements in the long term (6 months and 1 yr.). This behavior can be due to the acidic and hydrophilic character of the self-adhesive cements, which would lead to higher water sorption when compared to conventional hydrophobic resin cements. Therefore, perhaps the lower storage period observed in this study (90 days) was not sufficient to result in a significant difference between the groups, especially for the samples treated with chlorhexidine.

Regardless of the surface treatment (chlorhexidine) or storage period, there was a statistically significant difference between the self-adhesive cements used. As a result, the second hypothesis was validated. The different performances in the micro-shear test can be related to differences in their compositions, which in turn affect material properties such as elastic modulus, viscosity, and surface etching capacity [7, 14, 17]. Rely X U200 has a low initial pH (<2), which increases the potential for demineralization of this cement and seems to contribute to the higher bond strength values found in this study when compared with Smart Cem 2.

In this study, both luting agents showed lower bond strength values compared with conventional resin cements reported previously [5, 6, 11, 17]. This is due to four primary factors: (1) acidic monomers have low etching capacity, minimizing the surface demineralization; (2) the buffering effect of the minerals present in the dentin can neutralize the pH of the cement; (3) the high viscosity of the cements hinders their penetration into the interfibrillar space; (4) non-removal or incomplete removal of the smear layer promotes a weakly bonded reinforced resin intermediate layer [5, 6, 11, 16, 17].

Most of the groups showed adhesive failure, indicating lower resistance values between the self-adhesive cement and dentine. The loss of integrity of the resin-dentin interface during function is affected by thermal, mechanical, and chemical actions. These actions are detrimental to the longevity of indirect restorations luted with resin cements. More clinical trials are needed to assess the effects of the interaction between the self-adhesive resin cement and the use of chlorhexidine on bond durability.

Conclusion

It is concluded that regardless the material and storage period, the application of 2% chlorhexidine to dentin did not affect the bond strength of the self-adhesive cements tested.

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