Bond strength of a self-adhesive cement to deproteinized dentin: Effect of NaOCl concentration and application time

Resistência de união de um cimento resinoso auto-adesivo à dentina desproteinizada: efeito da concentração do hipoclorito e do tempo de aplicação

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Abstract

Purpose: This study investigated the effect of different protocols of deproteinization on the bond strength of a self-adhesive cement to dentin.

Methods: Buccal surfaces of bovine incisors were abraded to expose a flat dentin. Exposed dentin without any treatment was used as control 1, while the remaining specimens were acid-etched. After acid-etching and rinsing, the dentin was deproteinized using of following protocols: 5% NaOCl for 2 min, 5% NaOCl for 10 min, 20% NaOCl for 2 min, or 20% NaOCl for 10 min. The maintenance of wet dentin after rising of acid was used as control 2. Cylinders were formed using a self-adhesive resin cement RelyX Unicem and submitted to microshear test. Data were evaluated by one-way and two-way (excluding the controls) ANOVA and Tukey's test (α=0.05). Failure modes were classified under magnification.

Results: Only the deproteinization with 5% NaOCl for 10 min improved the bond strength compared to the absence of treatment (Control 1). Except for 20% NaOCl for 10 min, all protocols of deproteinization resulted in higher bond strength than those obtained to etched dentin (Control 2). Independently of application time, deproteinization with 5% NaOCl promoted higher bond strength than 20% NaOCl. It was observed a predominance of adhesive failures.

Conclusion: Both concentration and application time of NaOCl affected the bond strength of self-adhesive resin cement to deproteinized dentin.

Key words: Dental bonding; dentin; resin cements

Resumo

Objetivo: Avaliar o efeito de diferentes protocolos de desproteinização na resistência de união de um cimento auto-adesivo à dentina.

Metodologia: Superfícies linguais de incisivos bovinos foram desgastadas para expor uma dentina plana. Dentina exposta sem nenhum tratamento foi usada como controle 1, enquanto as amostras restantes foram condicionadas. Após condicionamento ácido e lavagem, a dentina foi desproteinizada usando um dos seguintes protocolos: NaOCl a 5% por 2 min, NaOCl 5% por 10 min, NaOCl a 20% por 2 min, ou NaOCl a 20% for 10 min. A manutenção da dentina úmida após a lavagem do ácido foi usado como controle 2. Cilindros foram construídos usando o cimento resinoso auto-adesivo RelyX Unicem e submetidos ao teste de micro-cisalhamento. Os dados foram avaliados por ANOVA um fator e dois fatores (excluindo os controles) e teste de Tukey (α=0,05). Os modos de falha forma classificados sob magnificação.

Resultados: Apenas a desproteinização com NaOCl a 5% por 10 min aumentou a resistência de união quando comparado a ausência de tratamento (Controle 1). Exceto para NaOCl a 20% por 10 min, todos os protocolos de desproteinização resultaram em maior resistência de união que aquela obtida na dentina condicionada (Controle 2). Independentemente do tempo de aplicação, a desproteinização com NaOCl a 5% promoveu maior resistência de união que NaOCl a 20%. Foi observada predominância de falhas adesivas.

Conclusão: Tanto a concentração como o tempo de aplicação interferem na resistência de união do cimento resinoso auto-adesivo à dentina desproteinizada.

Palavras-chave: Adesão dental; dentina; cimentos de resina

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Introduction

Successful bonding of luting materials to both restorative materials and dental substrate is essential in improving the performance of indirect restorations (1,2). Traditionally, adhesive cementation is a critical procedure that involves multiple steps, increasing the technical sensitivity (3,4). Thus, self-adhesive resin cements (SARCs) have been marketed to simplify clinical procedures and overcome this sensitivity of multi-step systems (5,6). SARCs do not require any pretreatment of the dental surfaces, and their application is performed by a single clinical step. However, in previous studies, SARCs demonstrated lower values of bond strength to dentin than conventional resin cements (7), while the previous use of adhesive system improved the bond strength values of SARCs (8).

One factor related to these lowest values of bond strength of SARC to dentin is the presence of smear layer (9). The smear layer impairs proper contact between SARCs and the underlying dentin during adhesive procedures (10-13). The mineralized components of the smear layer are efficient buffers, making the pH of the acidic monomers too high to demineralize the underlying dentin (9,14). Some solution acids has been applied previously to insertion of SARCs seeking to improve the bond strength to dentin (11-13). However, the results were not promisors. The previous etching with phosphoric acid before the placement of SARCs on the dentin substrate does not improve the bond strength since the presence of collagen fibril compromises the contact between the cement and the underlying dentin (9).

Previous study demonstrated that the deproteinzation with 5% NaOCl during 2 min was able to improve the bond strength of a SARC to dentin (15). However, it was demonstrated that the complete removal of the exposed collagen matrix from the etched dentin surface only can be achieved by applying solutions of around 10% NaOCl (16). However, this concentration requires a far longer than 10 min, which is not clinically acceptable. Thus, it is expected that higher concentrations of NaOCl than 10% can be able to remove the collagen mesh in shorter time.

This study aimed to evaluate the effect of concentration and application time of NaOCl on the bond strength of a SARC to deproteinzed dentin. The null hypothesis is that neither the application time nor the NaOCl concentration affects the bond strength.

Methodology

One week after extraction, sound bovine incisors were cleaned, polished, and examined under a light microscope in order to exclude any with cracks. Twenty four teeth were selected and stored in distilled water at 5°C for less than one month before the restorative procedure. Teeth were sectioned bellow the enamel-cement junction. Following, the crowns were sectioned parallel to the long axis into two halves (mesial and distal), resulting in 48 hemi-sections. The crown sectioning permitted to standard the dentin thickness. Afterwards, the buccal surface was wet-ground with 400-grit SiC abrasive papers until a flat surface in medium dentin was obtained (2 mm from the pulp chamber).

Each hemi-section was embedded in acrylic resin with the buccal/lingual face exposed. The dentin was wet-polished with 600-grit SiC abrasive papers to standardize the smear layer. Five specimens did not receive any treatment previously to restorative procedures (Control 1). The remaining specimens were etched with 34% phosphoric acid (3M ESPE, St. Paul, MN, USA) for 15 s, followed by rinsing with water for 10 s. Five of etched specimens were maintained wet after acid etching (Control 2). The remaining etched specimens were submitted to one of following protocols of deproteinization (n=5): 5% NaOCl for 2 min, 5% NaOCl for 10 min, 20% NaOCl for 2 min, or 20% NaOCl for 10 min. After deproteinization, the NaOCl was rinsed with water for 10 s and the dentin surface was air-dried with compressed air for 10 s.

Polyvinyl siloxane impression material molds (Aquasil Extra Low Viscosity, Dentsply DeTrey, Konstanz, Germany) presenting three cylindrical orifices [a cylinder-shaped orifice (1 mm inner diameter × 2 mm height)] were placed onto the dentin surface. The distances between the orifices were around 2 mm. The orifices were individually filled with a SARC, RelyX Unicem (3M ESPE, St. Paul, MN, USA) using an exploratory probe. The cement was light-cured for 20s using a light-emitting diode unit (Radii Cal; SDI, Bayswater, Victoria, Australia) with 800-mW/cm^2 irradiance. The distance between the mold and light-curing unit tip was set at around 1 mm during the light-activation. After the light-activation, the mold was removed to expose the cylinders, and the specimens were stored at 100% humidity for 24h.

The shear bond test was conducted using a universal testing machine (Instron 5565, Instron Canton, MA, USA). A thin steel wire (0.2 mm diameter) was looped around each cylinder and the shear load applied to the base of the cylinder at a crosshead speed of 0.5 mm/min until failure. Values of shear bond strength were converted to MPa using the following formula: MPa = Newtons/area (0.79 mm^2). The average value of the three bonded cylinders for each was recorded as the microshear bond strength (MPa) for that specimen. The data were subjected to one-way ANOVA and post-hoc Tukey's test (α=0.05). The data excluding that of the control groups were evaluated using two-way ANOVA (concentration x time) and post-hoc Tukey's test (α=0.05). After the bond test, the dentin surfaces were evaluated under optical microscopy at 40× magnification; failure modes were classified as adhesive, mixed, or cohesive failure within dentin.

Results

One way ANOVA (with control) showed significant effect of treatment (P<0.0001). The results of Tukey's test are displayed in Table 1. Only the deproteinization with 5% NaOCl for 10 min was effective in improving the bond

strength compared to the control without treatment. Except for the case of the application of 20% NaOCl for 10 min, the deproteinization of dentin increases the bond strength compared to dentin etched with phosphoric acid.

Table 1. Shear bond strength (MPa) for all experimental conditions (n=5).

Treatment of dentin substrate	Mean* (SD)
5% NaOCl for 10 min	18.3 (3.9) A
5% NaOCl for 2 min	17.2 (3.4) AB
20% NaOCl for 2 min	15.5 (3.8) ABC
Control 1 (absence of treatment)	13.1 (3.0) BCD
20% NaOCl for 10 min	11.7 (4.1) CD
Control 2 (only dentin-etching)	10.3 (1.2) D

* Significant differences between treatment of dentin substrate are indicated by distinct letters ($\alpha = 0.05$).

Two-way ANOVA (without control) showed significant effect only for the factor 'concentration of NaOCl' (P=0004). The factor 'application time' (P=0.325) and the interaction between the factors (P=0.077) were not significant. The results without controls are presented in Table 2. Independently of application time, the use of 5% NaOCl showed higher bond strength than 20% NaOCl. There was a predominance of adhesive failure for all experimental conditions.

Table 2. Shear bond strength (MPa) to deproteinized dentin according to protocols (application time x NaOCl concentration) of deproteinization (n=5).

NaOCl concentration	Application Time		Pooled average*
	2 min	10 min	
5%	17.2 (3.4)	18.3 (3.9)	17.7 (3.6) A
20%	15.5 (3.8)	11.7 (4.1)	13.6 (4.3) B

* For pooled average, means followed by distinct letters are significantly different (α=0.05).

Discussion

Only deproteinization with 5% NaOCl for 10 min was effective in improving the bond strength compared to the absence of treatment. Thus, the null hypotheses must be rejected. The main adhesive mechanism of SARCs is attributed to micromechanical retention and to the chemical reaction between phosphate methacrylates and hydroxyapatite (17,18). Thus, proper contact between the SARCs and the dentin is essential in improving the bond strength. Dentin etching with phosphoric acid did not improve the bonding of SARC to dentin. A previous study demonstrated that the creation of a thick and compact collagen mesh by acid etching prevents the viscous cement from reaching the deeper, unaffected dentin (9). Based on these previous findings, the removal of this collagen mesh seems to be important to improve the interaction between SARCs and the intact dentin.

Typically, the deproteinization of dentin is performed with 5% NaOCl for 2 minutes (15,19). This protocol was effective in improving the bond strength compared to dentin etched with phosphoric acid. However, there was no difference from the results for the absence of treatment, not justifying its use. A longer application time (10 min) improved the results and showed a significant difference from the controls. A possible explanation is that the prolonged time increases the collagen removal (16) and improves the contact between the SARC and the unaffected dentin. Clinically, however, the time of 10 min can be considered too long.

It was expected that a higher NaOCl concentration could compensate for a short application time. However, the use of 20% NaOCl resulted in lower bond strength values than 5% NaOCl for both application times. The NaOCl is a potent biologic oxidant and can inhibit the interfacial polymerization of resin-based materials (20,21). Thus, despite the possibly greater collagen removal, the use of NaOCl in higher concentrations can compromise a SARC's polymerization and reduce the bond strength. This possible negative effect seems to be increased by longer application times. In the present study, 20% NaOCl applied for 2 min resulted in higher bond strength than acid-etched dentin, while 20% NaOCl applied for 2 min showed similar values. The findings indicate that the deproteinization procedure can improve the bond strength of SARCs to dentin, while the use of a low-NaOCl solution for a longer period seems to be preferable.

Conclusions

Dentin deproteinization using low concentration of NaOCl for long time of application can be able to improve the bond strength of a self-adhesive resin cement.

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