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ORIGINAL ARTICLE

Residual effect of sodium hypochlorite on pulp chamber dentin adhesion

ABSTRACT

Aim: *This study has evaluated the residual effect of NaOCl on resin-pulp chamber dentin bond strength after 7 and 14 days, using a three-step etch-and-rinse adhesive system.*
Methodology: *Forty pulp chamber dentin from bovine incisors crowns were randomly allocated to one of the following groups: G1, immersion in 0.9% saline solution for 30 minutes (control); G2, immersion in 5.25% NaOCl for 30 minutes; G3, immersion in 5.25% NaOCl for 30 minutes and stored for 7 days; and G4, immersion in 5.25% NaOCl for 30 minutes and stored for 14 days. After restoration, the dentin/resin interface was tested by microtensile bond strength, and failure mode was analyzed by Scanning Electron Microscopy. Data were analyzed by ANOVA followed by Tukey.*
Results: *G1 had higher bond strength than the rest of the groups. There were no statistically significant differences among G2, G3, and G4 (p>0.05).*
Conclusions: *The adverse effect of NaOCl on bond strength persisted even after 14 days following exposure.*

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Introduction

A major goal of endodontic therapy is to eliminate microorganisms from the root canal system to create an environment that is most favorable for healing (1). This is accomplished as a result of mechanical cleaning and shaping along with irrigation using antibacterial agents (2). In order to keep the root canals free of contamination after endodontic treatment, a final restoration of the involved tooth should be performed with a careful technique (3). The infiltration of oral microorganisms in the root canal filling is favored by the lack of a proper coronary restoration, which impairs the prognosis of endodontic treatments (4). Likewise, restoration is important to preserve the remaining dental structure, avoiding fractures that could lead to tooth loss (5). Moreover, the restoration of endodontically treated teeth is critical for clinical success achievement (6-10).

One of the reasons for loss of adhesion of adhesive restorative materials and consequently an improper restoration may be the chemical substance used during the endodontic treatment irrigation (11-13). Sodium hypochlorite (NaOCl) is a widely used chemical irrigant for the biomechanical preparation of root canals due to its antimicrobial properties and capacity to dissolve organic matter (14). However, NaOCl can affect the structure of the dentin surface and degrade collagen fibers (12, 15). Sodium hypochlorite liberates chlorine in its cleavage (Cl_2) and oxidizing compounds; Cl_2 generates dissolution of collagen fibers and the oxidizing compounds interfere with the closing of the polymeric chain of the resin during the set time, causing incomplete polymerization and reduced adhesion to dentin. Once the bond of adhesive systems is due to the infiltration of resin monomers in the collagen layer, this degradation of the organic matrix will be a detrimental factor (16-19).

NaOCl is one of the most used substances for irrigation and decontamination during root canal treatment. Composite resin restorations have been used more and more worldwide. However, residual sodium hypochlorite in porosities of mineralized dentin may give rise to uncured resin polymerization, and thus undermine bond strength (20). The latter may explain a reduction in adhesive bond strength after its use. The aim of this study was to evaluate the residual effect of NaOCl on resin-pulp chamber dentin bond strength after 7 and 14 days, using a three-step etch-and-rinse adhesive system. There is a hypothesis that waiting 7 or 14 days to restore the teeth would not interfere in the reestablishment the bond strength between composite resin and pulp chamber dentin.

Materials and Methods

Forty bovine incisors were stored in 0.5% thymol and used within 2 months of extraction. Methods were performed by a single operator. The teeth were cut to expose the pulp chamber dentine of the middle third of the buccal part of the crown (3). Five mm was cut horizontally from the incisal portion of the crown with a double-sided diamond disc (KG Sorensen, Barueri, SP, Brazil) under refrigeration. Next, the middle third of the crown was removed at a height of approximately 8 mm corresponding to the double-sided diamond disc radius (KG Sorensen, Barueri, SP, Brazil), which then drilled into the middle third of the incisal border aligned to the long axis of the tooth, removing the buccal surface of the crown fragment. Once the samples were acquired, the pulp tissue was cautiously taken out with a spoon excavator. After, the intracoronary dentin was polished for 30 seconds with wet 180- and 600-grit silicon carbide abrasive paper under running water (Struers, Abramin, Copenhagen, Denmark) to plane and create a standardized smear layer. Forty rectangular dentin pieces were acquired.

Specimens were randomly allocated into four groups, with ten specimens in each group, according to the chemical irrigants used and time of storage, as follows: G1, immersion in 0.9% saline solution for 30 minutes (control); G2, immersion in 5.25% NaOCl for 30 minutes; G3, immersion in 5.25% NaOCl for 30 minutes and stored

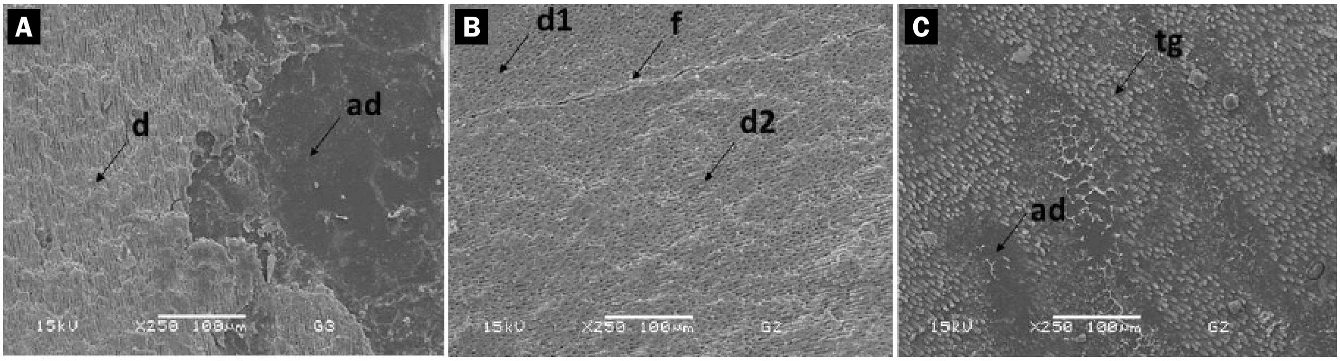

Figure 1
Scanning electron microscopic images of the different fracture patterns taken at 15 kV under x250 magnification. In **A**, a mixed fracture is noticed; it exhibits adhesive area (ad) and dentin (d). In **B**, it is shown the characteristic of two aspects of cohesive fracture in dentin (d1, d2), and the fracture line (f). And in **C**, both tags (tg) and fractured tags can be seen inside the adhesive matrix (ad), illustrating the adhesive failure.

for 7 days; and G4, immersion in 5.25% NaOCl for 30 minutes and stored for 14 days. During immersion, the samples were kept shaking, and the chemical substance was renewed every 10 minutes. Sample storage was done at 37 °C in a 100% humidified environment. After 14 days, 7 days, or immediately after immersion, according to each group, the adhesive system followed by composite resin (Scotchbond Multi Purpose and Filtek Z250, 3M ESPE, St Paul, MN, USA) were applied in the pulp chamber of all samples at the same day to avoid variants. Before the bonding procedures, all specimens were dried with absorbent papers and etched with 37% phosphoric acid for 15 seconds, rinsed for the same time, and dried with cotton balls. A total etching adhesive system was applied to the surface of the pulp chamber dentin according to the manufacturer's instructions. Three layers with 1 mm of a resin composite were incrementally added to the bonded dentin (21), and each one was light-cured for 20 seconds by using a halogen light-curing unit operating at 1200 mW/cm^2 (Optilight Max; Gnatus, Ribeirão Preto, SP, Brazil). After the composite filling of dentin, the blocks were stored in distilled water at 37 °C for 24 hours.

After 24 hours, specimens were removed from the water, dried, and fixed to an acrylic plate to allow the creation of serial cross-sections using a diamond saw operating at 300 rpm (Isomet; Buehler, Lake Bluff, IL). Twenty-five rectangular sticks (1±0.1 mm^2) were obtained from each group from the middle portion of the crown portion to ensure the existence of a linear resin/dentin interface. The sticks were individually attached to a testing apparatus [ie, the Geraldeli's device (22)], with a cyanoacrylate adhesive (Super-bonder Gel, Loctite Adesivos, Itapevi, SP, Brazil), and the dentin/resin interface was submitted to microtensile bond test in a universal testing machine (EZ Test, Shimadzu Co., Kyoto, Japan) at a crosshead speed of 1 mm/min until failure with 500N load cell adjustment.

The sticks cross sections were measured using a caliper (Vonder Digital Electronic Caliper, Curitiba, PR, Brazil) for calculating the bond area. The microtensile bond strength was determined and analyzed applying the analysis of variance and the Tukey test for post-hoc comparisons (α=0.05) using the BioEstat 2.0 program (CNPq 2000, Brasília, DF, Brazil). The failure modes were examined under a scanning electron microscopy (Figure 1) and classified into one of four types: adhesive (interfacial failure), adhesive/cohesive (mixed), cohesive in resin, and cohesive in dentin. The specimens were sputter-coated with gold in a Denton Vacuum Desk II Sputtering device (Denton Vacuum, Cherry Hill, NJ, USA) and observed by scanning electron microscopy (JSM/5600 LV - JEOL Ltd., Tokyo, Japan) operating at 15 kV.

Results

The microtensile bond strength means and standard deviations are presented in Table 1. Statistical analysis of the data revealed significant differences between pairs of means (P<0.05). The control group had the

highest bond strength compared to the other groups, although no statistically significant difference was observed between irrigation with 0.9% saline solution or 5.25% NaOCl in the immediately restored group (P>0.05). Moreover, no statistically significant differences were observed in irrigation among groups with 5.25% NaOCl (P>0.05).
Table 1 shows the failure modes observed in each group. All groups studied presented two or more failure modes and the predominating failure is the adhesive. The control group had a higher percentage of fractures in cohesive in resin than others, while the group irrigated with NaOCl and stored for 14 days was the only group with failure in dentin.

Discussion

The bond of therapeutic materials to dentin is a critical factor as it would avoid microleakage, favoring the results of the endodontic treatment (6). There are different restoration materials, such as amalgam, glass ionomer, and composite resin. Amalgam has been less used over time, as it has non-aesthetic characteristics, in addition to containing mercury in its composition. Composite resin has been the material of choice to restore endodontically treated teeth because of its hardness in comparison to glass ionomer, avoiding fracture of the teeth (23).
The use of chemical substances during endodontic treatment may interfere with the adaptation of the restorative material. A well-known interaction is the use of sodium hypochlorite with adhesive restorations (24). Sodium hypochlorite can decrease the bond strength of the adhesive materials to the dentin because it liberates oxidizing compounds and further degradation of the collagen fibers (20). However, it was unknown until the present study if, with time, this bond strength could be restored.
In this *in vitro* study, the coronal part of bovine teeth was used as a replacement for human teeth (25). The methodology was adapted (12) in order to analyze the bond strength of adhesive materials to the intracoronary dentin, where it is affected by chemical substances used during endodontic treatment. This is a unique study aimed to evaluate the bond strength of composite resin to the pulp chamber dentin after the use of sodium hypochlorite immediately, 7, and 14 days. We failed to reject the null hypothesis, which held that waiting 7 or 14 days to restore the teeth would not interfere to re-establish the bond strength between composite resin and pulp chamber dentin.
The results have shown there was no statistically significant difference between G1 and G2. We suggest there was not sufficient time to degrade the collagen fibers. However, G1 has had more cohesive in resin failures than the other groups, suggesting the control group as the one with the strongest bond strength.
There was also no statistically significant

Table 1
Bond strength mean and standard deviations (in MPa) and failure mode (%) according to the experimental groups

Group	Mean (Standard Deviation)	Failure Mode			
		Adhesive	Mixed	Cohesive in resin	Cohesive in dentin
Control	62.42 (37.74)[a]	44.1	14.7	41.2	0
0 days	48.10 (32.49)[a,b]	76.5	17.6	5.9	0
7 days	35.17 (26.66)[b]	54.3	37.1	8.6	0
14 days	35.57 (25.37)[b]	71.0	18.4	8.0	2.6

Same superscript letters in the column are not statistically significant (P>0.05).

difference among G2, G3, and G4 and it reveals the decreased bond strength generated by the sodium hypochlorite in accordance with the literature (24). Sodium hypochlorite can alter the mechanical features of the dentin, such as diminishing the flexural strength, elastic modulus, and hardness (26). Indeed, sodium hypochlorite is an oxidizing chemical substance that causes solid restraint of the interfacial polymerization of adhesive restorative materials (27). In this context, the reactive residual-free radicals from the sodium hypochlorite in the dentin compete with the propagating vinyl-free radicals from the resin light activation system, and consequently, a premature chain termination is formed along with an unfinished polymerization (18, 28).

We suggest that oxygen is not the only factor of this adverse effect on dentin since this effect would be restored after 14 days. Another study used 35% hydrogen peroxide bleached teeth, another oxidant agent, and was restored immediately and, also, after a week; they did not find a difference between the groups (29), which is in accordance with our study. Indeed, it could be that degradation of the collagen fibers also contributes to decreased bond strength, which because of the chlorine content, it breaks the bonds of the collagen fibers (15). The adverse effect of sodium hypochlorite continued to affect the dentin with time; this is believed by the type of fracture found in G4, cohesive in dentin, showing a weakness in the dentin structure. Hence, G1 and G2 have not shown a significant difference.

EDTA (ethylenediaminetetraacetic acid) is a chelating agent used in the field of Endodontics (30). It is able to demineralize the dentin and to rearrange the collagen structure after its use (31). However, after some time of the use of the EDTA, remineralization can occur (31). When EDTA is used after NaOCl, it is able to improve the bond strength (18, 32). EDTA might increases bond strength due to its anti-oxidant power via redox reaction, allowing for free radicals polymerization without premature chains or failures (18).

Alteration of the adhesive system to the sodium hypochlorite could prompt in higher bond strength. However, in this research, the adverse effect of this irrigating solution persisted even after 14 days. It should be due to the slow liberation of the oxidizing agents released by the sodium hypochlorite, persisting in a high amount at the dentinal structure. The degradation that occurred in the dentin is not re-established by the time. The literature demonstrates that sodium thiosulfate is able to restore the dentin bond strength after the use of sodium hypochlorite, allowing the restoration of the tooth immediately after the irrigation protocol (24). Researches seeking to restore collagen fibers or a deep investigation in the antioxidant agents are needed since it would be more favorable to restore the tooth immediately after endodontic treatment.

Conclusions

Within the limitations of this study, the adverse effect of NaOCl on bond strength persisted even after 14 days of its exposure.

Clinical Relevance

The restoration of endodontically treated teeth is critical for clinical achievement. This study showed that restoring the teeth immediately after NaOCl irrigation resulted in not significantly different bond strength than irrigating with saline. However, restoring the teeth 7 or 14 days after NaOCl irrigation resulted in significantly reduced bond strength.

Conflict of Interest

The authors deny any conflicts of interest related to this study.

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