Immediate Shear Bond Strength of Resin Cements to Sodium Hypochlorite–treated Dentin

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Abstract

Introduction: The purpose of this *in vitro* study was to evaluate the immediate shear bond strength of different categories of resin cements on sodium hypochlorite (NaOCl)-treated dentin and to evaluate if the bond was improved by a subsequent treatment with 10% sodium ascorbate before adhesive procedures. **Methods:** This study tested immediate shear bond strengths to human dentin of 5 resin cements: Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein), Multilink (Ivoclar Vivadent), Clearfil Esthetic Cement EX (Kuraray, Tokyo, Japan), SpeedCEM (Ivoclar Vivadent), and Clearfil SA Cement (Kuraray). All cements were tested with no NaOCl pretreatment of the dentin (negative control) and with a 20-minute exposure of the dentin to 6% NaOCl before bonding procedures. The cements found to have decreased bond strengths to NaOCl-treated dentin were tested with the dentin exposed to 10% sodium ascorbate after NaOCl exposure. The sodium ascorbate exposure times tested were 5 seconds and 1 minute. **Results:** The mean and standard deviation values for immediate shear bond strength (MPa) for the negative control group were as follows: Variolink II, 18.8 $\pm$ 4.2; Multilink, 29.1 $\pm$ 7.1; Clearfil Esthetic Cement EX, 20.7 $\pm$ 4.9; SpeedCEM, 17.8 $\pm$ 4.2; and Clearfil SA Cement, 7.2 $\pm$ 2.8. The results for the NaOCl exposure group were as follows: Variolink II, 24.0 $\pm$ 6.7; Multilink, 34.1 $\pm$ 6.1; Clearfil Esthetic Cement EX, 20.7 $\pm$ 6.8; SpeedCEM, 0.0 $\pm$ 0.0; and Clearfil SA Cement, 0.1 $\pm$ 0.1. The results for the 5-second sodium ascorbate group were the following: SpeedCEM, 8.5 $\pm$ 2.6, and Clearfil SA Cement, 4.3 $\pm$ 2.0. The following results were found for the 1-minute sodium ascorbate group: SpeedCEM, 12.2 $\pm$ 3.2, and Clearfil SA Cement, 4.8 $\pm$ 1.0. **Conclusions:** The resin cements tested varied in their capacity to adhere to NaOCl-treated dentin. Some resin cements exhibited equal or improved bond strengths ($P < .05$), whereas others exhibited significantly decreased bond strengths ($P < .05$). For the susceptible resin cements, a rinse of 10% sodium ascorbate provided an immediate restoration of at least 50% of the original bond strength ($P < .05$). The efficacy of sodium ascorbate may vary among resin cements. *(J Endod 2014;40:1459–1462)*

Key Words

Ascorbic acid, dentin bonding, resin cements, root canal irrigants, shear strength, sodium hypochlorite

The use of adhesive procedures in the restoration of endodontically treated teeth (ETT) is common. Adhesively retained composite cores, composite restorations to close access openings, and resin-cemented posts have become mainstay treatment options (1, 2). Advances in treatment modalities have led to the successful restoration of ETT with minimal to no mechanical retention with adhesively cemented monolithic all-ceramic full-coverage restorations (3). Some of these ceramic restorations are without traditional retention and resistance form, requiring adhesive bonding with a resin cement to be successful.

The application of sodium hypochlorite (NaOCl) to dentin during endodontic treatment may affect dentin bonding negatively. Several studies have found that an exposure time of 15–20 minutes significantly reduces dentin bond strengths (4–7). It is unclear whether the effects of NaOCl exposure are universal to all bonding systems and/or chemistries (8, 9). Several studies have documented improved dentin bond strengths after NaOCl exposure by application of sodium ascorbate, even to the point of complete reversal (8,10–13).

One purpose of this *in vitro* study was to evaluate the immediate shear bond strength of resin cements on NaOCl-treated dentin. The second purpose of the study was to evaluate if a subsequent treatment with 10% sodium ascorbate before adhesive procedures improves the bond strength.

Materials and Methods

Preparation of Specimens

Seventy noncarious, nonrestored human molars and premolars were stored for no more than 3 months in a solution of 0.5% Chloramine T Trihydrate (Fisher Scientific, Pittsburgh, PA) at room temperature. After sectioning each tooth through the central fissure in a mesial to distal direction, each half of the sectioned tooth was embedded in cold cure acrylic (ProBase; Ivoclar Vivadent, Schaan, Liechtenstein) with the sectioned dentin surface exposed for bonding. Each exposed dentin surface was prepared for testing with 600-grit sand paper and stored in room temperature tap water until use. Specimens were prepared and used on the same day.

Method of Testing

One end of 140 ceramic rods (eMax Press, Ivoclar Vivadent) was etched with 4.9% HFl acid (IPS Etching Gel, Ivoclar Vivadent) for 20 seconds, rinsed, dried, and silanated

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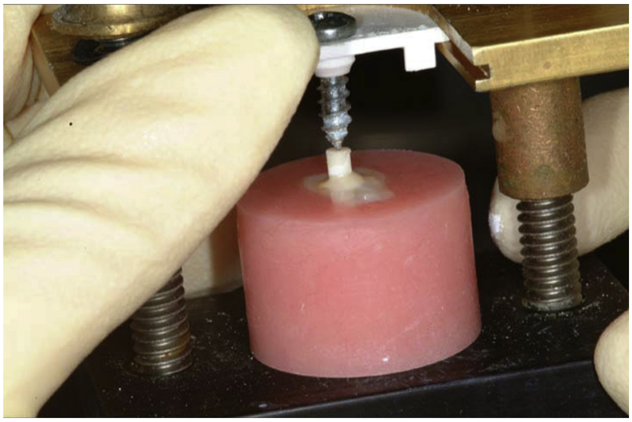

Figure 1. Rod stabilization in an Ultradent jig.

for 1 minute with Monobond Plus (Ivoclar Vivadent). Dentin bonding surfaces were blotted dry with a Kimwipe (Kimberly Clark, Irving, TX) immediately before the bonding procedures. Dentin surfaces were treated per manufacturer instructions for each cement. The etched and silanated end of the ceramic rod was copiously covered with the cement being tested and placed on the exposed dentin surface of a single specimen. The rod was placed solely in dentin, no more than 2 mm away from the dentinoenamel junction. The specimen was placed into an Ultradent Jig (Ultradent Products Inc, South Jordan, UT), and the rod was stabilized (Fig. 1). Excess cement was removed from around the rod with microbrushes. Each specimen was cured with a Blue Phase G2 curing light (Ivoclar Vivadent) on the high setting for 20 seconds from the front of the jig and 20 seconds from the back, placing the light at a distance of 1 mm from the ceramic rod. The specimens were then immediately taken for shear bond testing (Instron Machine; Instron, Norwood, MA). Testing was performed at a crosshead speed of 1 mm/min. All specimens were subjected to shear bond testing within 5 minutes of completion of the bonding procedures.

Resin Cements Tested

Five resin cements were evaluated: Variolink II with Excite F (Ivoclar Vivadent), Multilink (Ivoclar Vivadent), Clearfil Esthetic Cement EX with Clearfil DC Bond (Kuraray, Tokyo, Japan), SpeedCEM (Ivoclar Vivadent), and Clearfil SA Cement (Kuraray). Cements were categorized according to the type of bonding sequence (ie, total etch, self-etch, self-etch, and self-adhesive).

Cement type 1, requiring a total-etch technique, consisted of Variolink II. Cement type 2, requiring the application of a self-etching, self-adhesive dentin-bonding agent, consisted of Multilink and Clearfil Esthetic Cement EX. Cement type 3 (ie, self-etching, self-adhesive cements) consisted of SpeedCEM and Clearfil SA Cement.

Experimental Groups

Each experimental group (Table 1) consisted of 10 specimens for each resin cement tested. Each cement was initially bonded to dentin that was not pretreated with NaOCl to serve as a negative control group for the specific cement.

Each cement was tested a second time for immediate shear bond strengths on dentin surfaces that had been exposed to 6% NaOCl (Vista Dental Products, Racine, WI) for 20 minutes. The length of exposure time and concentration of NaOCl were intended to simulate clinical conditions for a tooth undergoing an endodontic procedure. Before NaOCl exposure, each specimen was fitted with a watertight plastic coping; 2 mL 6% NaOCl was placed within each coping, submerging the exposed dentin surface in 2–3 mm of NaOCl. Specimens were kept partially submerged in a 37°C water bath for 20 minutes to emulate physiological temperature conditions. The specimens were removed from the bath and rinsed, the plastic copings were removed, and the exposed dentin surfaces were rinsed with water for 5 seconds. The specimens were blotted dry with a Kimwipe and immediately subjected to the shear bond test protocol.

The cements that showed a decrease in bond strength on NaOCl-treated dentin were tested with 2 additional dentin treatment protocols to evaluate the potential reversal effects of treating the dentin with sodium ascorbate after NaOCl treatment. In the first test sequence, specimens were exposed to 6% NaOCl for 20 minutes as previously described. After plastic coping removal and rinsing with water, each exposed dentin surface was rinsed for 5 seconds with 1.2 mL 10% sodium ascorbate (Fisher Scientific, Pittsburgh, PA) followed by 5 seconds of water rinsing and blotted dry with a Kimwipe. Cements were used according to the manufacturer's instructions to bond ceramic rods to the treated dentin surface as described previously. The specimens were then subjected to the shear bond strength protocol.

In the second test sequence, specimens underwent 20 minutes of 6% NaOCl exposure. Before plastic coping removal, the NaOCl was discarded, and the exposed dentin surface was rinsed twice with water and the water discarded. The exposed dentin surface was blotted dry with a Kimwipe; 2 mL 10% sodium ascorbate was placed in the plastic coping, submerging the exposed dentin surface. After 1 minute, the sodium ascorbate was discarded, the plastic copings were removed, and the dentin surfaces were rinsed with water for 5 seconds. The surfaces were blotted dry with a Kimwipe, and the specimens were subjected to the shear bond strength protocol.

Results

The mean values and standard deviations are listed in Table 2. Statistical analysis of data was performed with analysis of variance and Tukey post hoc tests. Cement type 1 exhibited greater bond strengths when the dentin was pretreated with NaOCl ($P < .05$). The immediate shear bond strengths for cement type 2 were unaffected by NaOCl pretreatment ($P < .05$). Bond strengths for cement type 3 were significantly

TABLE 1. Resin Cements Evaluated

Cements	Bonding steps required		Experimental groups		
	Phosphoric acid etch	Dentin bonding agent	Negative control	NaOCl	NaOCl + sodium ascorbate
Variolink II	Yes	Yes	Yes	Yes	No
Multilink	No	Yes	Yes	Yes	No
Clearfil Esthetic Cement EX	No	Yes	Yes	Yes	No
SpeedCEM	No	No	Yes	Yes	Yes
Clearfil SA Cement	No	No	Yes	Yes	Yes

TABLE 2. Testing Results (mean values ± standard deviation)

Immediate shear bond strength (MPa)	Negative control	6% NaOCl for 20 minutes	Sodium ascorbate for 5 seconds	Sodium ascorbate for 1 minute
Variolink II	18.8 ± 4.2	24.0 ± 6.7	—	—
Multilink	29.1 ± 7.1	34.1 ± 6.1	—	—
Clearfil Esthetic Cement EX	20.7 ± 4.9	20.7 ± 6.8	—	—
SpeedCEM	17.8 ± 4.2	0.0 ± 0.0	8.5 ± 2.6	12.1 ± 3.2
Clearfil SA Cement	7.2 ± 2.8	0.1 ± 0.1	4.3 ± 2.0	4.8 ± 1.0

less to NaOCl pretreated dentin than the negative control group ($P < .05$). Treatment with sodium ascorbate restored approximately 50% of immediate shear bond strengths to dentin for cement type 3, with no statistical differences between a 5-second and 1-minute exposure time to sodium ascorbate ($P < .05$).

Discussion

There are conflicting reports in the literature as to how the exposure of dentin to NaOCl affects subsequent dentin bond strengths. The resin cements in this study that used a total etch technique or a separate self-etching primer and bonding agent before cement bonding were either unaffected or their bond strengths were significantly greater after a 20-minute exposure to 6% NaOCl. A previous study using a total etch concept and Excite F as a dentin bonding agent also found no negative effects from NaOCl exposure (8). This is in contrast with other studies using different etch and rinse systems, which found a decrease in dentin bond strength after NaOCl exposure (8, 10, 12). Although 1 previous study using a self-etching, self-adhesive bonding agent found no decrease in bond strength after NaOCl pretreatment (9), several others have found decreased bond strengths for the adhesive systems they tested (6, 8, 10, 12). Only the self-etching, self-adhesive cements in this investigation showed decreased bond strengths after NaOCl exposure. Another study also found a self-etching, self-adhesive resin cement to be susceptible to NaOCl (12).

Weston et al (11) postulated that compromised bonding in NaOCl-treated teeth is a result of the removal of organic matrix from the dentin by the NaOCl, leaving a less receptive bonding surface (11). They also proposed that NaOCl oxidizes a component in the dentinal matrix that interferes with free radical propagation at the resin-dentin interface during adhesive procedures. This reduction in bond strength does not appear to improve over time (14). Several studies have successfully reversed this process with the application of sodium ascorbate (8,10–13). Several authors have suggested that this improvement in bond strengths is the result of a redox reaction in which the sodium ascorbate or other acids changes the oxidized dentin back to a reduced substrate (8, 11, 13). Weston et al (11) hypothesized that the mechanism by which the reversal occurs may even be a near instantaneous process.

The use of a reducing agent to reverse the effects of NaOCl on susceptible bonding systems is an effective solution to restore bond strengths when bonding to ETT with the tested systems. Further research is needed on the effects of these reducing agents on long-term dentin bond degradation. Erhardt et al (15) found that sodium ascorbate may compromise or enhance the degradation of dentin bonding over time depending on the adhesive system used. It should be noted that in that study the reducing agent was added to the adhesive, not applied and rinsed off as in other studies.

This study found that a 5-second rinse with 10% sodium ascorbate reversed the effects of NaOCl as well as a 1-minute rinse, supporting Weston et al's hypothesis of a near instantaneous reversal. This is in contrast to the findings of Prasansuttipron et al (13), who found a 5-second rinse of sodium ascorbate to be ineffective. None of the test groups in the present study exhibited a complete reversal of dentin bond strengths as seen in other studies (8,10–13). Although previous studies examined microtensile bond strengths at 24 hours, this study tested immediate shear bond strengths. One limitation of this study is that the NaOCl was not refreshed during the 20-minute exposure period as it is during an endodontic procedure. Refreshing the NaOCl may have allowed for further oxidation of the dentin by NaOCl and resulted in different bond strength results.

Conclusions

The 5 resin cements tested in this study vary in their immediate shear bond strength to NaOCl-treated dentin. The total etch systems and systems with a separate dentin bonding agent were not negatively affected by previous NaOCl exposure. The self-etching, self-adhesive resin cements were adversely affected by NaOCl exposure. For those susceptible resin cements, a rinse of 10% sodium ascorbate provided an immediate restoration of at least 50% of the original bond strength. The efficacy of sodium ascorbate may vary among bonding systems.

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