Effect of NaClO Treatment on Bonding to Root Canal Dentin Using a New Evaluation Method

Tomoyasu ISHIZUKA, Hiroki KATAOKA, Takatomo YOSHIOKA, Hideaki SUDA, Naohiko IWASAKI, Hidekazu TAKAHASHI and Fumio NISHIMURA

Pulp Biology and Endodontics,  
 Oral Biomaterials, Department of Restorative Sciences, Graduate School, Tokyo Medical and Dental University  
 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

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The purposes of this study were to investigate the reliability and efficiency of a new evaluation method for resin bonding to root canal dentin, which measures both marginal adaptation and shear bond strength simultaneously, and to determine the effects of root canal irrigants on resin bonding. A wet bonding system (Single Bond) and a self-etching primer system (Clearfil Mega Bond) were employed; NaClO was used as a root canal irrigant. No gaps or changes in bond strength were observed despite the NaClO treatment when the wet bonding system was employed, while the gap formation ratio increased, and bond strength decreased with longer NaClO treatment time when the self-etching primer system was employed. These findings suggested that this new experimental method was effective for evaluating resin systems to the root canal wall dentin which is affected by irrigation with NaClO.

Key words: Adhesive resin, Dentin, Adhesion test

INTRODUCTION

Recent studies reported that endodontically treated teeth are not reinforced by a full-coverage crown combined with post and core systems. Those studies stated that preservation of the tooth structure is an important factor to prolong the longevity of the tooth. In addition, minimizing the amount of tooth structure loss is reported to be essential for a favorable prognosis. However, a good performance of the resin bonding system to the tooth structure is desired to restore the tooth after root canal treatment.

The reliability and predictability of resin bonding to enamel are well established. However, those to dentin are still questionable because dentin has a greater organic content than enamel. Moreover, both the composition and morphology of deep dentin are different from those of superficial dentin. For example, the hardness of deep dentin is less than that of superficial dentin; the density and diameter of dentinal tubules of deep dentin are greater and thicker, respectively, compared with superficial dentin; the calcification of deep dentin is not as mature as that of superficial dentin. Therefore, bond strength to deep dentin has been reported to be lower than that to superficial dentin. In addition, deep dentin and root canal wall dentin...
might be affected by root canal irrigants and disinfectants during endodontic treatment. NaClO is one of the most common root canal irrigants used for debridement, lubrication, destruction of microbes and dissolution of organic tissues\(^8\). There have been several studies reporting the effects of NaClO on resin bonding systems. Some reported that NaClO treatment interfered with the adhesive of the bonding system to dentin\(^9,10\) but others reported that NaClO treatment improved the adhesion of the bonding system to dentin when using phosphoric acid\(^11\). Therefore, the effects of NaClO treatment on dentin have not clearly been confirmed.

Recently, we contrived a new evaluation method for resin bonding to root canal wall dentin, which measures both marginal adaptation and shear bond strength simultaneously. In the present study, we investigated the reliability and efficiency of this new method in an effort to examine the effects of root canal irrigants on resin bonding to dentin.

MATERIALS AND METHODS

Experimental teeth and resin bonding systems
One hundred extracted bovine mandibular first and second incisors were used. They were frozen at \(-29^\circ\mathrm{C}\) immediately after extraction and thawed before use. Two dentin bonding systems, a wet bonding system (Single Bond, 3M, St Paul, MN, USA; SB) and a self-etching primer system (Clearfil Mega Bond, Kuraray, Osaka, Japan; MB), were employed as shown in Table 1.

Cavity preparation
The crown of the tooth was removed with a diamond disk at the cement-enamel junction. The pulp tissue was removed using a dental file (#30, Maillefer, Ballaigues, Switzerland) without root canal preparation. Each root was embedded in the center of an acrylic pipe (diameter: 16 mm, depth: 25 mm) with dental stone (New Fuji Rock, GC, Tokyo, Japan; W/P=10 ml/50 g). A 3 mm thick coronal surface was resected with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Then, the root canal space was filled with a thermoplasticized gutta-percha delivery.

Table 1 Bonding systems used

<table>
<thead>
<tr>
<th>Product name</th>
<th>Code</th>
<th>Manufacturer</th>
<th>Conditioner</th>
<th>Primer</th>
<th>Bonding resin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single Bond</td>
<td>SB</td>
<td>3M, St. Paul, MN, USA</td>
<td>35% H(_3)PO(_4) (9BB)</td>
<td>—</td>
<td>Vitrabond copolymer, Bis-GMA, HEMA, PI, Ethanol, Water (9DR)</td>
</tr>
<tr>
<td>Clearfil Mega Bond</td>
<td>MB</td>
<td>Kuraray, Osaka, JAPAN</td>
<td>MDP, HEMA, Water, PI, Methacrylate (13A)</td>
<td>—</td>
<td>MDP, HEMA, Methacrylate, PI, Filler (22A)</td>
</tr>
</tbody>
</table>

Abbreviations; MDP: methacyryloyloxydecyl phosphoric acid, PI: Photo initiator
HEMA: 2-hydroxyethylmethacrylate, Bis-GMA: Bisphenol-glycidyl methacrylate
\(\square\): Batch No
EVALUATION OF BONDING TO ROOT CANAL DENTIN

system (Obtura II, Obtura, Fenton, Mo, USA) without any endodontic sealers. A truncated cone cavity (4 mm in base diameter, 2.5 mm in depth and a convergence angle of 5°), was prepared at the top portion of the root canal using a drilling machine (ASD-360, Ashina, Hiroshima, Japan) and a tapered end mill (B-12, OSG, Nagoya, Japan). All cavities were rinsed with distilled water using a three-way syringe (Fig. 1).

Preparation of surface for adhesion
A 6 v/v% NaClO solution (Purelox, Oyalox, Tokyo, Japan) was selected as a root canal irrigant to examine its effects on bonding systems. After a designated period of NaClO treatment, each cavity was rinsed with distilled water for 10s and one of the aforementioned dentin bonding systems was applied according to the manufacturer’s instructions (Table 2).

The following five dentin preparations were performed (Table 3): A, neither NaClO treatment nor bonding system; B, bonding system without NaClO treatment; C, NaClO for 1 min and bonding system; D, NaClO for 5 min and bonding system; E, NaClO for 10 min and bonding system.

After dentin conditioning, a composite resin (Clearfil AP-X A3.5, Kuraray,

![Diagram](image_url)

Fig. 1 Preparation of specimen.
The tooth was embedded in the center of an acrylic pipe. After filling the root canal with gutta-percha, a truncated cone cavity was prepared at the top portion of the root canal.

Table 2 Bonding procedures of the two bonding systems

<table>
<thead>
<tr>
<th>SB</th>
<th>1. Application of etchant to dentin for 15 seconds.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2. Rinsing and blotting excess of water leaving the tooth moist.</td>
</tr>
<tr>
<td></td>
<td>3. Application of adhesive, drying gently for 2-5 seconds, light-curing for 10 seconds.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>MB</th>
<th>1. Dentin conditioning and priming with primer for 20 seconds.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2. Drying with mild air.</td>
</tr>
<tr>
<td></td>
<td>3. Application of bond, drying with mild air, light-curing for 10 seconds.</td>
</tr>
</tbody>
</table>
Table 3 Conditions for the adhesive surface

<table>
<thead>
<tr>
<th>Condition</th>
<th>6% NaClO</th>
<th>Bonding system</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>-</td>
<td>+</td>
</tr>
<tr>
<td>C</td>
<td>1 min</td>
<td>+</td>
</tr>
<tr>
<td>D</td>
<td>5 min</td>
<td>+</td>
</tr>
<tr>
<td>E</td>
<td>10 min</td>
<td>+</td>
</tr>
</tbody>
</table>

Fig. 2 Evaluation of gaps.

A specimen in which India ink penetration was observed anywhere around the margin was defined as contraction gap positive.

Osaka, Japan) was filled in the cavity and photo-polymerized using a light-curing unit (New Light VL-11, GC, Tokyo, Japan) for 40s. Ten specimens were prepared for the two bonding systems in each group.

Observation of contraction gap

Immediately after the resin filling, the tooth surface in the acrylic pipe was ground with a series of sheets of silicon carbide paper (#320, #500 and #1000) under distilled water. Subsequently, India ink (Bokuju, Kaimei, Tokyo, Japan) was spread on the ground surface for 5s and washed out with distilled water for 10s. Marginal adaptation of the resin was investigated with a CCD camera (VH-6110, Keyence, Osaka, Japan) at 27 x magnification. When India ink penetration was observed anywhere around the margin of a specimen, it was defined as contraction gap positive (Fig. 2).

Measurement of punch out shear bond strength

After the contraction gap observation, all specimens were kept in distilled water at 37°C for 24h. A superficial 1.0 mm coronal layer of each specimen was sectioned with a low-speed diamond saw. The thickness of the sectioned specimen and the diameter of the resin-filled cavity were measured using a digital micrometer (MDC-25, Mitsutoyo, Tokyo, Japan) with 0.001 mm precision and a measuring device installed.
in the CCD camera with approximately 0.04 mm precision, respectively. The sectioned specimen was positioned into a brass jig for the punch out shear bond strength test (Fig. 3). This jig consisted of two parts; the upper part had a cylindrical projection at the center (3.9 mm in diameter, 3.0 mm in height), and the lower part had a round hole (4.0 mm in diameter) also at the center. The load was applied from the apical side of the specimen. The initial dislodgement load by which the filled resin was punched out from the root canal wall cavity was measured using a universal testing machine (1123, Instron, Canton, MA, USA) at a crosshead speed of 1.0 mm/min.

The punch out shear bond strength was defined as the dislodgement load divided by the bonded area (approximately 13 mm²).

Statistic analyses
Occurrence of the gap formation was analyzed using the chi square test at a significance level of 5%. The punch out shear bond strength was evaluated using the one-way ANOVA and the Tukey-Kramer multiple range test at a 5% significance.

RESULTS
Results of contraction gap measurements for SB and MB are illustrated in Figs. 4 and 5, respectively. All specimens of condition A showed marginal gaps. In contrast, neither the specimens of B, C, D and E in which SB was applied nor those of B in which MB was applied, showed any gaps. However, specimens of C, D and E, in which MB was applied, showed gaps significantly more frequently with longer NaClO treatment time.

Results of the punch out shear bond strength are shown in Figs. 6 and 7. The error bars indicate standard deviations. All specimens of condition A showed poor shear bond strengths. The bond strengths of SB for specimens of B, C, D and E were 7.1, 8.1, 10.8 and 9.8 MPa, respectively. There were no significant differences among those values. However, the bond strengths of MB for B, C, D and E were 5.6,
4.0, 2.3, 1.2 MPa, respectively. There were no significant differences between B and C, between C and D and between A, D and E.

**DISCUSSION**

There are only a few studies\(^\text{12-14}\) that evaluated resin bonding efficiency to the root canal wall dentin. In these studies, the flat dentin surface sectioned along the root canal was prepared. In the present study, the cavity was prepared around the root canal dentin wall and the bonding systems were applied from the pulpal side. In addition, almost all directions of the dentinal tubules were perpendicular to the cavity wall. The convergence angle of the prepared cavity was 5°, which was similar to that of a tapered fissure-bar for dowel space preparation. Therefore, the cavity preparation used was considered to be typical for composite resin restoration after
root canal treatment.

Perfect marginal adaptation of a composite resin to dentin is essential to avoid marginal discoloration and leakage. There are several methods to evaluate marginal adaptation, such as microleakage test and wall-to-wall gap observation in ISO/TR11405: 1994 (15). In the present study, the observation method of the contraction gap similar to ISO/TR11405: 1994 was employed. India ink, a colloidal solution of carbon particles, was applied for visualizing the marginal gap. Its penetration is considered to provide a similar rank order to sealing ability of bacterial ingress and to be a potentially suitable tracer for microleakage/dentin permeability assessment (16).

The tensile bond or shear bond test is generally employed to evaluate the resin bonding strength to dentin. However, the punch out shear bond test has been suggested to be more effective to evaluate resin bonding to dentin (17). In addition, the experimental set-up newly devised by us, could measure both marginal adaptation and bond strength using the same specimen. Moreover, it was possible to apply the load perpendicular to the cavity wall which consisted of root canal wall dentin. Therefore, this method could be employed to evaluate adhesion of luting materials for dowel post systems to the root canal wall. However, there are a few disadvantages in this method. For example, specimen preparation is complicated, and the results are influenced by the elastic property of the filling materials used; punch out bond strength is increased when a filling material with a low elastic modulus is employed because the filling material deforms during loading, which interferes with the pushout from the cavity. Therefore, truncated cone cavities were employed in the present study to avoid interference caused by the filled material’s deformation.

The configuration of cavity preparation is an influential factor for wall-to-wall gap formation. Recently, the “c-factor” was introduced to determine the cavity configuration effect on resin bond strength to dentin (7,18). The apical side of the prepared cavity was faced with gutta-percha which did not adhere to the composite resin in the present study. Therefore, the c-factor of the prepared cavity was calculated to be approximately 1.25; the value was similar to a class II or III cavity, which might be favorable for resin bonding to dentin (18).

The dentin bonding systems used in the present study represent the wet bonding system (SB) and self-etching primer system (MB). In the wet bonding system, the etchant removes the smear layer and demineralizes dentin, and the adhesive resin penetrates the etched dentin. However, the acid component of the self-etching primer mildly demineralizes dentin so that it does not remove smear plugs completely. At the same time, the primer component modifies the demineralized dentin and the bonding resin infiltrates the primed dentin. Therefore, the conditions of dentin before applying bonding resin in the two bonding systems employed in the present study were different.

Several studies reported on the marginal adaptation and bond strengths of SB and MB. Marginal gap formation of SB was observed in 10-30% of specimens despite strict observance of the manufacturer’s instructions (19,20). In the present study, however, no gaps were observed in either SB or MB. The direction of dentinal tu-
bules perpendicular to the cavity wall might enhance the bonding strength of the SB because the resin tags are resistant to contraction shrinkage due to polymerization. Bond strengths of SB and MB were reported to be 4.8-20.1 MPa and 15.7-30.5 MPa, respectively. In the present study, bond strength was lower than in previous studies, because those studies applied bonding systems to flat dentin surfaces and c-factor was smaller than in the present study.

There are many studies that evaluated the relationship between marginal adaptation and bond strength. Some studies have suggested little correlation between bond strength and marginal adaptation, while others suggested that higher tensile bond strength meant lower dye microleakage. In the present study, the punch out bond strength did not always show a smaller value even when marginal gaps were observed. The scatter diagram showing the relationship between the punch out bond strength and gap free ratio of MB is shown in Fig. 8. The gap free ratio increased with the bond strength up to 5.6 MPa. The bonding strength of SB, more than 5.6 MPa, showed 100% gap free ratio. These findings suggest that the marginal gap may not be produced when the bond strength is greater than a certain value; the bond strength of 5.6 MPa was considered to be the threshold for gap formation of the composite resin and the adhesive systems employed in this study.

Recently, there have been several studies reporting the effects of endodontic irrigants on resin bonding systems. In particular, the effects of NaClO have been thoroughly discussed but not clearly confirmed. Dentin is degenerated by NaClO treatment because of the dissolution of dentinal collagen. Moreover, residual NaClO may interfere with polymerization of the bonding resin due to oxygen generation. The bond strength of resin following NaClO treatment before etching decreased when a MMA-TBB resin system was employed. The decreased bond strength is improved when an ascorbic acid or a sodium thiosulfate solution is applied after NaClO treatment. These solutions remove NaClO by the oxidation-reduction reaction. Nikaido et al. evaluated the bonding strength at the buccal dentin surface after NaClO treatment on root canal wall dentin; the bonding strength of SB was significantly de-
increased after NaClO treatment, while that of a self-etching primer system did not change. In the present study, the bonding strength of MB decreased following NaClO treatment whereas that of SB did not change. Therefore, the effect of NaClO treatment on bonding strength may depend on the location of NaClO application. The self-etching primer of MB might not be effective for removing degenerated dentin and residual NaClO, while the etchant of SB might be strong enough to remove both. The remained smear layer after self-etching primer application might prevent oxygen from penetrating through dentinal tubules from the root canal wall dentin, while the removal of the smear layer by the etchant of SB treatment might allow oxygen penetration. The presence of oxygen may interfere with the polymerization of the bonding resin. Moreover, the effects of NaClO might be different between superficial dentin and root canal wall dentin. These may explain the discrepancy between the present findings and those previously reported.

However, the findings of this study were obtained after a relatively short water storage period without thermal stress. It is necessary to evaluate the changes of marginal adaptation and bond strength using a longer water storage period under thermal stress.

In conclusion, this new experimental design for examining the marginal adaptation and bond strength was considered to be effective for evaluating resin bonding efficiency to root canal dentin. Using this method, we found that NaClO interferes with the bonding ability of MB but does not affect that of SB.

REFERENCES