Adhesion of resin core foundation composites to root canal dentin using different dentin adhesives

PENPAPA WATTANASUKCHAI, DDS, RANGSIMA SAKOOLNAMARKA, DDS, PhD & MANSUANG ARKSONNUKIT, DDS, MS, PhD

ABSTRACT: Purpose: To evaluate the microtensile bond strengths (μTBS) of two core composites; LuxaCore and MultiCore Flow, to root canal dentin when using different adhesive systems. Methods: Bonded specimens were allocated into six groups (N= 20) according to the combination of two core composites and three adhesive systems (All-Bond 2, Excite DSC and Contax). The materials were used according to the manufacturers’ instructions. The μTBS were measured using a universal testing machine with a crosshead speed of 1 mm/minute. Data were analyzed utilizing two-way ANOVA (α= 0.05). Results: Excite DSC showed the highest mean μTBS, which was significantly different from those of All-Bond 2 and Contax in LuxaCore groups. Excite DSC showed the highest μTBS but significant differences (P= 0.0154) were not detected in MultiCore groups. There were significant differences in μTBS between two resin composites (P< 0.001). With Contax, μTBS of MultiCore Flow to dentin were significantly higher than those of LuxaCore (P= 0.003). (Am J Dent 2010;23:98-102).

CLINICAL SIGNIFICANCE: Adhesive systems affected the adhesion of LuxaCore–Automix Dual to root canal dentin. However, there was no effect on MultiCore Flow. For the two core composites used in this study, Excite DSC apparently showed better adhesion than the others.

Introduction

Increased emphasis is being placed on the conservation of natural tooth structure, and the use of bonding technologies permits a more conservative approach to post and core restorations. Recently, the utilization of fiber-reinforced composite posts in combination with adhesive resin cement to restore endodontically treated teeth has increased in popularity. In a flared canal, an excessively thick layer of resin cement in the coronal region of the post space could occur. This layer of resin cement may not be strong enough to resist occlusal loading. To overcome this problem, a dual-cure resin composite has been introduced as a luting medium, because it has a higher modulus of elasticity than resin cement and has a modulus of elasticity close to dentin and fiber posts.¹

Currently, clinicians can use various “etch-and-rinse” or “self-etching primer” adhesive systems for bonding to root canal dentin. The etch-and-rinse technique followed by a hydrophilic adhesive system has been shown to provide better bond strength to occlusal dentin.² For etch-and-rinse systems, the degree of drying of the exposed collagen after etching is critical, since excessive drying will result in exposed collagen fibrils collapsing, thus preventing the penetration of resin monomer and thus a lower bond strength.³ Self-etching primer systems are generally less technique-sensitive because such systems simultaneously demineralize the dentin and infiltrate resin monomer into the collagen,⁴ which eliminates the rinsing and drying steps. However, most studies are on coronal dentin, and little is known about bonding to root canal dentin.

Dual-cure adhesives are generally used for bonding to root canal dentin, because of their ability to self-polymerize in the absence of light, especially in the deeper regions of a post cavity. However, an adverse chemical reaction has been reported between chemically-activated resin composite and acidic resin monomers. Self-etching primer contains an acidic monomer, and consequently a high concentration of uncurable acidic monomers will be present within the primed dentin surface.⁵ Therefore, the compatibility of dual-cure adhesive and self-etch primed dentin is questionable.

This study evaluated the μTBS of two dual-cured resin composite core materials to root canal dentin using three dentin adhesives. The null hypothesis was that there were no differences of the μTBS between resin composites and root canal dentin using different dentin adhesives.

Materials and Methods

Preparation of bonded specimens - The Ethics Committee of the Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand approved the study according to the protocol in compliance with the International Conference on Harmonization/Good Clinical Practice (ICH/GCP). Thirty single-rooted human premolar teeth, recently extracted from adolescents for orthodontic reasons and stored in 0.1% thymol solution for no longer than 6 months, were decoronated at the cement-enamel junction using a low speed cutting machine (Isomet⁶). Pulpal tissue was removed using endodontic files and the post spaces were prepared using reamers (Peeso reamers⁷) in a low-speed handpiece under copious water to a depth of 5 mm and with an ovoid shape 3 mm long and 2 mm wide. After post space preparation, the root canals were rinsed with 2.5% sodium hypochlorite solution 10 mL and sterile isotonic sodium chloride solution 10 mL and dried with paper points. The external surfaces of the roots were covered with light-activated resin composite to make grips for testing, and wrapped in aluminum foil. The chemical compositions of materials are presented in Table 1.

The roots were randomly divided into six groups, each consisting of five specimens, and their root canal dentin surfaces treated with one of the following bonding agents following the manufacturers’ instructions: All-Bond 2, Excite DSC⁸ or Contax.⁹ The post spaces were filled with one of the
Table 1. Materials composition.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Core materials</td>
<td></td>
</tr>
<tr>
<td>LuxaCore-Automix Dual</td>
<td>Barium glass and pyrogenic silica, Bis-GMA</td>
</tr>
<tr>
<td>MultiCore Flow</td>
<td>Dimethacrylates, barium glass, Ba-Al-fluorescent-silicate glass, ytterbium trifluoride, catalysts, stabilizers and pigments</td>
</tr>
<tr>
<td>Bonding agents</td>
<td></td>
</tr>
<tr>
<td>All-Bond 2 (3-step etch-and-rinse system)</td>
<td>Primer A: NTG-GMA (2.1%), acetone, ethanol, water. Primer B: BPDM (24%), acetone, ethanol, photoinitiator. D/E resin: Bis-GMA, HEMA, photoinitiator (CQ), amine activator. Pre-bond resin: Bis-GMA, HEMA, benzoyl peroxide.</td>
</tr>
<tr>
<td>Bis-GMA, Bisphenol glycidyl methacrylate; NTG-GMA, N-tolylglycine glycidyl methacrylate; BPDM, Biphenyl dimethacrylate; HEMA, 2-Hydroxyethyl methacrylate; CQ, Camphoroquinone</td>
<td></td>
</tr>
</tbody>
</table>

![Illustration of the bonding and μTBS test procedures.](image)

The mean μTBS and standard deviations for all groups are presented in Table 2. Two-way ANOVA in Table 3 showed significant differences in μTBS for resin composites (P < 0.001) and dentin adhesives (P = 0.001). However, the interaction between resin composites and dentin adhesives was not significant (P = 0.469).

The μTBS of LuxaCore with Excite DSC to dentin were significantly higher than those of LuxaCore groups; All-Bond 2 and Contax. On the other hand, μTBS of MultiCore Flow with Excite DSC to dentin were not significantly higher than...
Fig. 2. SEM images of the fractured surfaces from LuxaCore groups showing adhesive failure. Arrows show resin tags completely peeled off from the dentin tubules from LuxaCore with All-Bond 2 (A), whereas the specimens from LuxaCore with Excite DSC failed in the hybrid layer which show collagen mesh (arrows) (B). LuxaCore with Contax, arrows show irregular smear plugs in the dentin tubules (C). (Magnification x5000).

Fig. 3. SEM images of the fractured surfaces from MultiCore Flow groups showing adhesive failure. Resin tags had peeled off the dentin tubules, arrows show slightly rougher surfaces from partial collagen mesh from MultiCore Flow with All-Bond 2 (A), whereas the failed surfaces from MultiCore Flow with Excite DSC happened in the hybrid layer, arrows show collagen fibers around resin tags (B). MultiCore Flow with Contax, arrows show irregularly shaped resin tags surrounded by some collagens (C). (Magnification x5000)

Table 4 demonstrated the percentage of fracture modes for each experimental group. Fracture analysis indicated that the majority of failures were adhesive (Figs. 2, 3). There were no significant differences between the failure modes in each group.

SEMs of the fractured surfaces of specimens from the LuxaCore groups and the MultiCore Flow groups with three types of dentin adhesives are shown in Figs. 2 and 3. The MultiCore Flow specimens showed surfaces of resin tags with collagen mesh that allowed more infiltration of the dentin adhesives into the etched dentin.

In the present study, the $\mu$TBS was evaluated only in the coronal region of the root canal. The reason for this is because the adhesion between resin composite and dentin occurs mostly at this region. The thickest region of the resin composite in the root canal is the coronal one-third region. The space in the middle and apical two-thirds region is available only for the post and adhesive materials. Many studies have compared the regional bond strengths in the root canal. However, the results are controversial. It was suggested that the nature of dentin surface of the canal wall or the tubule density might not be attributable to the difference in bond strengths between coronal and middle/apical regions.

Published research on the bonding of resin composite core materials to root canal dentin is extremely limited. The present study revealed that the $\mu$TBS of LuxaCore to dentin with Excite DSC was significantly higher than that of LuxaCore groups with All-Bond 2 and Contax. The $\mu$TBS of MultiCore Flow to dentin with Excite DSC was not higher than that using other bonding agents. Previous studies showed higher bond strengths than in the current study. Aksornmuang et al. used Nano-Bond, a two-step self-etch adhesive system similar to Contax, but their mean bond strength was 47.46 MPa which was higher than the current finding of 33.8 MPa for Contax. This may be because their post space had not been exposed to sodium hypochlorite (NaOCl). NaOCl has been extensively used in endodontic therapy to provide gross debridement, disinfection, lubrication, and dissolution of tissues. In this experiment, the post spaces were prepared in root canals which had not been endodontically treated, but had been exposed to NaOCl in order to simulate the clinical situation. This powerful antimicrobial
agent has been previously shown to jeopardize the polymerization of bonding resins.\textsuperscript{13,14} It was hypothesized that NaOCl led to oxidation of some component in dentin matrix, forming protein-derived radicals\textsuperscript{15} that would compete with the propagating vinyl free-radicals generated by the light activation of resin adhesives, resulting in premature chain termination and incomplete polymerization.\textsuperscript{15} It has also been reported\textsuperscript{14,17} that the bond strength to root canal dentin is affected by the chemical irrigant, depending on the type of bonding agent. In agreement with our findings, the \( \mu \text{TBS} \) to dentin in root canals were lower than other studies\textsuperscript{11,13} that did not expose root canal dentin to NaOCl.

The higher \( \mu \text{TBS} \) using Excite DSC compared to All-Bond 2, both being etch-and-rinse adhesive systems, may arise from the different solvents used. All-Bond 2 comprises a primer with acetone as the solvent which is easier to evaporate than the ethanol used for the Excite DSC primer solvent. Acetone is used in bonding agents in order to increase their hydrophilicity and lower the viscosity. However, acetone evaporates much more quickly than ethanol, so All-Bond 2 may lose its effectiveness especially for root canal dentin. On the other hand, ethanol evaporates more slowly than acetone, and therefore may penetrate better and more deeply into dentin in the root canal. This may result in complete impregnation of the adhesive resin into the collagen of the demineralized dentin, which is a necessary requirement for high bond strengths.\textsuperscript{19} The difficulty in controlling the surface hydration of a deep and narrow post space may affect the \( \mu \text{TBS} \) of LuxaCore, using All-Bond 2 as a bonding agent, which was significantly lower than when using Excite DSC. SEM observation (Fig. 2A) revealed that the surfaces around the resin tags of LuxaCore with All-Bond 2 were flat, which represented collapsed collagen fibers and thus incomplete infiltration of the bonding agents and compromised hybrid layer formation. LuxaCore with Excite DSC (Fig. 2B) showed surfaces of resin tags with collagen mesh that allow complete infiltration of the bonding agents into the etched dentin. Excite DSC is a 2-step etch-and-rinse system, which combines priming and bonding to simplify the bonding procedure. Excite DSC, which incorporates catalyst into the bristles of the application brush, may be easier to use compared to All-Bond 2 when applying bonding agents to the dentin, which is difficult to access within the root canal.

Contax self-etching primer system which is less technique-sensitive than an etch-and-rinse system, simultaneously demineralizes and infiltrates resin monomer into the collagen with no rinsing and drying steps.\textsuperscript{20} However, it has been assumed that in order to achieve the hybrid layer and to create retention when resin cement or bonding agent is used,\textsuperscript{21} it is necessary to remove the smear layer and debris from the dentin canal walls and the initial part of dentin tubules. The \( \mu \text{TBS} \) of Luxacore with Contax to dentin was significantly lower than with Excite DSC. This may reflect the better removal of the smear layer and debris and cleaning of the dentin surface when using Excite DSC, which is supported by the SEM images (Fig. 2C).

It is also interesting to note that MultiCore Flow had a higher \( \mu \text{TBS} \) irrespective of bonding system, compared to LuxaCore. Both are dual-curing, radiopaque resin composites specifically designed for the fabrication of core build-ups and the cementation of root canal posts. LuxaCore consists of barium glass and pyrogenic silica in a Bis-GMA-based matrix. The filler is 72% by weight and the filler size is 0.02-4 \( \mu \text{m} \). MultiCore Flow has a matrix of Bis-GMA, UDMA and TEGDMA totaling 29% by weight. The inorganic fillers are barium glass, ytterbium trifluoride, barium aluminum fluorosilicate glass and highly dispersed silicon dioxide. The filler volume is 70% by weight and the filler size is 0.04-25 \( \mu \text{m} \). The difference in their monomer matrix compositions may be the reason why MultiCore Flow showed higher \( \mu \text{TBS} \), regardless of bonding system, compared to LuxaCore. LuxaCore is composed primarily of Bis-GMA, which has been shown to have a lower conversion level when it is not mixed with low viscosity resins.\textsuperscript{22,23} SEM images (Fig. 3A-C) revealed that the surfaces of the resin tags of MultiCore Flow specimens with all three dentin adhesives were different from the LuxaCore specimens (Fig. 2A-C). The MultiCore Flow specimens showed surfaces of resin tags with collagen mesh that allow more infiltration of the dentin adhesives into the etched dentin. MultiCore Flow showed better bond strengths to any dentin adhesives tested in this study. Higher bond strength was achieved when using Excite DSC as a dentin adhesive for both resin composites tested, but statistically significant differences were only detected with the LuxaCore group (\( P<0.05 \)). These high bond strengths of MultiCore Flow with Excite DSC might be due to the chemical compatibility of the products produced by the same manufacturer. From the results, the null hypothesis was rejected because there were differences between the \( \mu \text{TBS} \) of three dentin adhesives when used with the same resin composite; LuxaCore groups, and between two resin composites when used with the same dentin adhesive; Contax.

Within the limitations of this study, it can be concluded that different types of dentin adhesives affect the \( \mu \text{TBS} \) between LuxaCore and root canal dentin. Excite DSC, a "2-step etch-and-rinse" adhesive system showed significantly high \( \mu \text{TBS} \) in LuxaCore.

\begin{itemize}
\item a. Buehler Ltd. Lake Bluff, IL, USA.
\item b. Dentsply, DeTrey, Zurich, Switzerland.
\item c. DMG, Hamburg, Germany.
\item d. Ivoclar-Vivadent, Schaan, Liechtenstein.
\item e. Bisco, Schaumberg, IL, USA.
\item f. 3M ESPE, St. Paul, MN, USA.
\item g. Lloyd Instrument, Bucks, UK.
\item h. Dentsply, Milford, DE, USA.
\item i. JEOL, Tokyo, Japan.
\item j. SPSS Inc, Chicago, IL, USA.
\item k. Pentron Clinical Technologies, Wallingford, CT, USA.
\end{itemize}

Acknowledgements: The authors gratefully acknowledge the financial support received from the Faculty of Dentistry, Chulalongkorn University and the National Research Council of Thailand (NRCT), and Professor Martin Tyas, University of Melbourne, Australia, for assistance in preparation of the manuscript.

Disclosure statement: Authors have no conflict of interest.

Dr. Wattanasukchai is a graduate student, Dr. Sakoolnamara is Assistant Professor, Department of Operative Dentistry, and Dr. Arksornnukit is Associate Professor Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand.

References

1. Boschian Pest L, Cavalli G, Bertani P, Gagliani M. Adhesive post-endodontic restorations with fiber posts: Push-out tests and SEM observa-


