Abstract: The study evaluated the influence of different luting materials on the microtensile bond strength of glass fiber posts to root canal dentin. Thirty extracted maxillary premolars were endodontically treated, and the roots were prepared for post cementation using the FRC Postec system (Vivadent). Two luting materials (Multilink, Vivadent and Clearfil Photo Core, Kuraray) were used in combination with three adhesive: Multilink Primer (Vivadent), Clearfil Photo Bond, and Clearfil New Bond (Kuraray). A composite build-up was performed around the root to provide adequate gripping during testing. Specimens were cut to obtain beams with the post in the center and with the radicular dentin overlaid by the composite build-up on each side. Microtensile testing was performed with a universal testing machine at a cross-head speed of 0.5 mm/min. The failure mode was classified under a stereomicroscope and four representative beams of each group were selected for SEM analysis. Bond strength data that were analyzed with two-way ANOVA and Student-Newman-Keuls multiple comparisons tests revealed that adhesive systems, luting materials, and the interaction between these two factors significantly influenced the bond strength results ($p<0.01$). Multilink applied with its own adhesive system obtained the best results, while the lowest bond strength was achieved with clearfil photo core in combination with multilink primer.


Keywords: fiber post; microtensile test; self-etching adhesive; radicular dentin; curing mode

INTRODUCTION

Fiber posts are becoming increasingly popular for the restoration of endodontically treated teeth. They provide retention for core restorations when the coronal portions of the teeth exhibited a severe loss of tooth substances.1–4

The retention of fiber posts within root canals is affected by several factors involving the type of the post, its adaptation to the post space, and the luting agent.5,6 Resin-based luting agents in combination with dentin adhesives are commonly used for the cementation of fiber posts.7,8 The rationale for using fiber posts for the rehabilitation of endodontically treated teeth is to create a restoration with an elastic modulus that is close to dentin, and to produce stress fields that are similar to those experienced by natural teeth during occlusal loading.3,9,10 The clinical results are predictable when fiber posts are closely adapted to root canal spaces, with the canal walls being surrounded by a thin and uniform film of cement.9,11,12

A range of results were reported when different commercially available dentin adhesive and luting cement combinations were employed for cementing fiber posts.8,13,14 These materials may polymerize through a light-activated reaction, a chemical reaction or a combination of both mechanisms.15 Recent investigations suggested the possibility of adverse interactions between the polymerization modes of simplified dentin adhesives and chemical-cured resin composites that may affect the bond integrity.16,17

The observation of imperfectly round root canal cross sections after endodontic treatment is not uncommon.18,19 The presence of a discrepancy between the post and the shaped canal is considered an indication for using resin
composites to lute or reline the post to improve its fit and retention.\textsuperscript{9,11}

The objectives of this investigation were: (1) to compare the performance of conventional resin cement and a resin composite and (2) to evaluate the influence of different combinations of adhesive systems and luting agents when fiber posts were luted to prepared root canals. Evaluation was performed using the microtensile bond strength test. The null hypothesis tested was that the bond strength of fiber posts to intraradicular dentin is not affected by the different combinations of dentin adhesives and luting cements.

**MATERIALS AND METHODS**

Thirty human maxillary premolars, extracted for orthodontic or periodontal reasons, were selected for the study. The teeth were stored in 1\% Chloramine T solution at 37°C, to prevent bacterial growth, until their use. The crown of each tooth was removed 1 mm coronal to the cemento-enamel junction, with a water-cooled diamond blade (Accutome-50, Struers, Copenhagen, Denmark). The root canals were instrumented according to a step-back technique. Manual instrumentation was performed to ISO size 35, using a series of stainless steel K-files to the working length of each tooth. The rest of the canal was prepared mechanically with Gates-Glidden drills that were used in sequence from No. 2 to No. 4 (Union Broach, New York, USA). The root canals were irrigated with 5.25\% NaOCl at 37°C and 10\% EDTA solution alternately and dried with multiple paper points. All the teeth were obturated with a cold lateral compaction technique, using gutta-percha cones and a zinc oxide eugenol-based root canal sealer (Pulp Canal Sealer, Kerr, Romulus, MI, USA). The access to the root canal was filled with a provisional filling material (Cavit, 3M Espe, St.Paul, MN, USA).

The endodontically treated teeth were stored in deionized water at 37°C for 24 h prior to the preparation of post spaces. The coronal gutta-percha was removed from the root canal, with a Largo Drill, and a post space was prepared (FRC Reamer size No.1, Ivoclar-Vivadent, Schaan, Liechtenstein), leaving 5 mm of gutta-percha to preserve the apical seal. Translucent glass fiber posts (FRC Postec size 1 batch no. 94017, Ivoclar-Vivadent) were used. They are made of glass fibers that are embedded in a dimethacrylate matrix. Each post was fitted to the post space and cut at adequate length.

The teeth were randomly divided into six groups of five teeth each, according to the different material selected for the luting procedure. In group A, Multilink (ML) (batch no.00072, Ivoclar-Vivadent), a self-curing resin luting agent was used. For group B, Clearfil Photo Core (CPC) (batch no. 510A Kuraray Medical Inc., Tokyo, Japan), a light-curing composite material, was selected. Three adhesive systems were applied in combination with the two luting resin composite materials, was selected. Evaluation was performed using the microtensile bond strength test. The null hypothesis tested was that the bond strength of fiber posts to intraradicular dentin is not affected by the different combinations of dentin adhesives and luting cements.

A pH meter was used for pH measurement of the adhesives employed (Micro pH 2000, Crison Instruments, Alella, Spain). Before performing the luting procedure, the surface of each post was silanized with Monobond-S (Batch no. F50602, Ivoclar-Vivadent) and gently air-dried after 60 s. Monobond-S is a pre-hydrolyzed 3-methacryloxypropyl-tri-methoxysilane (3-MPS) in a water/ethanol solvent. The luting procedures were performed as described in Table I. The resinous material was inserted into the root canal, with a lentulo drill.

After post insertion, composite core build-up was performed on each tooth at the coronal level, to avoid the risk of coronal leakage and to provide a sufficient bulk for handling. A light-curing composite material was used for subgroups A (Tetric Ceram, Ivoclar-Vivadent), while the same CPC was applied following an incremental technique on specimens of subgroup B. Each increment was polymerized for 20 s (output 600 mW/cm\textsuperscript{2}, Optilux, Demetron, Sybron-Kerr, Orange, CA, USA).

**Microtensile Bond Strength Test**

Specimens were stored in deionized water at 37°C for 24 h, before testing. To provide an adequate gripping on the loading machine, additional composite build-up was made on the external root surface, following a technique previously described by Boulliaguet et al.\textsuperscript{15} Briefly, the outer surface of each root was etched with 37\% phosphoric acid for 15 s, rinsed with water, and gently air-dried. An adhesive system (Single Bond, 3M Espe) was applied, air-thinned, and light-cured for 20 s (Optilux, Demetron). The bulk of composite was performed with a light-curing composite following an incremental technique (Tetric Ceram). Each increment was polymerized for 20 s.

Each specimen was sectioned perpendicularly to its longitudinal axis into 0.8-mm-thick slabs, with a diamond blade, under continuous water cooling (Accutome-50, Struers, Copenhagen, Denmark). Each slab was then transversally sectioned at the outermost periphery of the post so as to obtain beams of ~1 mm\textsuperscript{2} of area, the diameter of which was measured with a digital caliper (Mituyoto, Tokyo, Japan).

Each stick was attached with Zapit (Dental Ventures of America, Corona USA) to the flat grip of a Bencor Multi-T testing assembly (Danville Engineering, San Ramon, CA) and loaded in tension at a cross-head speed of 0.5 mm/min until failure, using an universal testing machine (Instron Model 4411, Instron, Canton MA, USA).

Fractured specimens were examined at 40\times magnification under a stereomicroscope (Olympus SZ-CTV, Olympus, Tokyo, Japan) to determine the failure mode. Failures were classified as adhesive (at the post/cement or cement/dentin interface), cohesive (in the cement or dentin), or mixed.
The bond strength data were statistically analyzed with two-way ANOVA, to evaluate the performances of the different luting materials and the interaction between adhesive systems and the luting agent. Multiple comparisons were performed with Student-Newman-Keuls test. Statistical significance was set at $p < 0.05$.

### Scanning Electron Microscopy Analysis

Four fractured beams of each group that were classified as mixed failures were prepared for scanning electron microscopy (SEM) examination. Specimens were rinsed with 96% ethanol (Sigma, Aldrich Chemic, GmbH, Steinheim, Germany), air-dried, sputter-coated with gold (Polaron Equipment Ltd., Newhaven, England), and observed under an SEM at different magnifications (DSM-950, Zeiss, Germany).

### RESULTS

#### Microtensile Bond Strength Test

Mean microtensile bond strength values are shown in Table II. Two-way ANOVA showed a statistically significant influence of the adhesive system ($p < 0.0001$), and the luting agent ($p < 0.01$) on the bond strength results. The interaction between these two variables was also significant ($p < 0.01$). Student-Newman-Keuls multiple comparisons test revealed that when using CPC, all adhesives performed similarly. Clearfil photo bond and Clearfil New Bond exhibited lower bond strengths when applied in combination with ML cement. Multilink Primer performed better when applied in combination with its own luting cement. Acidic pH values were obtained from all the adhesive systems. Clearfil New Bond and CPC had similar pH values (2.55 and 2.52 respectively), while Multilink Primer recorded a lower value (1.95).

### Microscopic Evaluation

The distribution and percentages of failures are described in Table III. Most of the recorded failures were adhesive in nature and occurred predominantly along the adhesive/dentin interface. In some groups, a higher incidence of mixed failure was registered. SEM examination revealed different fracture patterns in the tested groups. Some specimens showed a

### TABLE I. Mode of Application and Composition of Tested Materials

<table>
<thead>
<tr>
<th>Materials</th>
<th>Components</th>
<th>Mode/Steps of Application</th>
</tr>
</thead>
<tbody>
<tr>
<td>Multilink (Ivoclar Vivadent)</td>
<td>Base and catalyst: DMA, HEMA, inorganic fillers, ytterbium trifluoride, initiators, stabilizers, and pigments</td>
<td>Mix primer A and primer B and apply for 15 s. Gently air dry. Mix the cement and apply on the teeth. Remove cement excess immediately. Wait for 120 s.</td>
</tr>
<tr>
<td>Primer A: Aqueous solution of initiators (sulfonate, amines)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Primer B: Phosphonic acid acrylate, HEMA, TEGDMA, methacrylate modified polyacrylic acid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Clearfil Photo Core (Kuraray)</td>
<td>Silanated silica, silanated barium glass, bisphenol A diglycidylmethacrylate, CQ</td>
<td>Apply on the restoration. Light cure for 40 s.</td>
</tr>
<tr>
<td>Clearfil Photo Bond (Kuraray)</td>
<td>K-etchant gel Catalyst liquid: Bisphenol A diglycidyl methacrylate, 10-MDP, HEMA, hydrophobic dimethacrylate, benzoyl peroxide, CQ Universal liquid: $N,N'$-Diethanol p-toluidine, sodium benzene sulfinate, ethyl alcohol</td>
<td>Etch for 15 s. Rinse with water spray and gently dry with air and paper points. Mix catalyst and universal liquid. Apply with a brush. Gently air dry for 2–3 s. Light cure for 10 s.</td>
</tr>
<tr>
<td>Clearfil New Bond (Kuraray)</td>
<td>K-etchant gel Catalyst liquid: Bisphenol A diglycidyl methacrylate, 10-MDP, HEMA, hydrophobic dimethacrylate, benzoyl peroxide Universal liquid: $N,N'$-Diethanol p-toluidine, sodium benzene sulfinate, ethyl alcohol</td>
<td>Etch for 15 s. Rinse with water spray for 10 s. Mix catalyst and universal liquid. Apply with disposable brush. Dry gently for 2 or 3 s.</td>
</tr>
</tbody>
</table>

DMA: dimethacrylate; HEMA: hydroxyethyl methacrylate; TEGDMA: triethylene glycol-dimethacrylate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; CQ: Camphorquinone.

### TABLE II. Mean (Standard Deviation) of Microtensile Bond Strength Values (MPa) Obtained for Each Tested Group

<table>
<thead>
<tr>
<th>Tested Group</th>
<th>Clearfil Photo Core</th>
<th>Multilink Cement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Clearfil Photo Bond</td>
<td>11.89 (4.34) 1a</td>
<td>11.37 (5.20) 1b</td>
</tr>
<tr>
<td>Clearfil New Bond</td>
<td>13.02 (3.39) 1a</td>
<td>9.57 (1.34) 2b</td>
</tr>
<tr>
<td>Multilink Primer</td>
<td>10.75 (2.95) 1a</td>
<td>15.33 (1.95) 2a</td>
</tr>
</tbody>
</table>

Letters show differences within the same column and numbers within the same row ($p < 0.05$).
complete detachment of the luting cement from the intraradicular dentin; residuals of adhesives were only present within the tubules [Figure 1(a)]. Others were characterized by the presence of a partial detachment of the adhesive and overlying cement on the post surface, especially when ML was applied as the luting cement (Figure 2).

### DISCUSSION

In this study, the microtensile bond strength test was used for evaluating the adhesive strengths of glass fiber posts in the root canals, as more information could be obtained compared with “push-out” or “pull-out” test, which were employed traditionally for assessing post retention. As the microtensile bond strengths of the dentin adhesives were significantly different when they were used in combination with the resin-based luting cements, we have to reject the null hypothesis that the bond strength of fiber posts to intraradicular dentin is not affected by the different combinations of dentin adhesives and luting cements.

The adhesive systems selected in this study were based on two different bonding strategies: (1) Clearfil Photo Bond and New Bond are simplified etch-and-rinse adhesives that require etching and rinsing with phosphoric acid prior to bonding. Being an ionic resin monomer with acidic functional groups, 10-MDP readily diffuses into the exposed collagen fibrils of the demineralized intraradicular dentin in the absence of smear layer. Poor control of moisture and incomplete resin infiltration may affect their efficacy; (2) Multilink Primer is a simplified self-etch adhesive, and it is directly applied on the smear layer and, due to its acidic pH (1.93), it etched thorough the smear layer and partially demineralized the underlying intact dentin.

When Clearfil Photo Bond was applied to the post space, tensile bond strength was not affected by the choice of luting cements. Being a dual-curing adhesive system, the initiators that catalyze its setting reaction may not have unfavorable interaction with both luting cements. Conversely, the bond strengths of clearfil new bond were reduced when it was

<p>| TABLE III. Distribution (%) of Failure Modes as Observed with Optical Microscopy |
|----------------------------------|----------------|----------------|----------------|</p>
<table>
<thead>
<tr>
<th>Clearfil Photo Bond</th>
<th>Clearfil New Bond</th>
<th>Multilink Primer</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mixed</td>
<td>33</td>
<td>50</td>
</tr>
<tr>
<td>Post/cement</td>
<td>25</td>
<td>20</td>
</tr>
<tr>
<td>Dentin/cement</td>
<td>42</td>
<td>30</td>
</tr>
</tbody>
</table>

| Multilink Primer                 |               |                |                |
|----------------------------------|---------------|---------------|
| Mixed                            | 25            | 23            | 47             |
| Post/cement                      | 25            | 31            | 12             |
| Dentin/cement                    | 50            | 46            | 41             |

Figure 1. Representative SEM images of the fracture pattern on dentin. A: A complete exposition of the dentinal substrate with adhesive remnants at the tubular level (×1000, bar = 20 μm). B: A partial detachment of the luting cement from dentin were registered in the tested groups (×3000, bar = 10 μm).

Figure 2. Microphotograph of the post surface after testing. A thin layer of residual resin cement remained on the post surface (×3000, bar = 10 μm).
applied in conjunction with the self-curing luting cement (ML). The adhesive polymerized through a characteristic slow-setting chemical reaction; this aspect could be considered a favorable condition for reducing stress at the bonding interface. Some chemical incompatibility probably exists and/or impurities (commonly water) may have penetrated the interface between the adhesive and the resin cement, affecting polymerization. Recent studies showed that the bonding efficacy of simplified etch-and-rinse adhesives to auto-cured composites/cements is hampered by the intrinsic permeability of these adhesives to water as a result of their increased hydrophilicity. 

This phenomenon has been shown to occur in vivo in bonded vital crown dentin and recently in endodontically treated teeth. The absence of differences in moisture content between a vital and a nonvital tooth and the reduction of dentin thickness, due to the preparation of the dowel space, may account for this intrinsic permeability. Rinsing with water during the etching procedure, especially in narrow elliptic root canals, combined with the presence of hydrophilic monomers in the adhesives, probably resulted in the retention of remnant water within the dentinal tubules, which, in turn, may affect the bond quality. Chemically cured composites polymerize more slowly than light-cured composites, allowing sufficient time for water to diffuse through the polymerized, simplified adhesives. This poor permeability of these adhesives to water as a result of their increased hydrophilicity, has partially reduced the shrinkage stress at dentin inter-

The command cure of the composite that was induced by light activation may have prevented the diffusion of water through the adhesive layer, interfering with adhesion. In general, the amount of water movement across resin-bonded dentin when etch-and-rinse adhesives are used is greater than that with self-etching adhesives. The adhesive layer may have partially reduced the shrinkage stress at dentin interface. As a result, a higher percentage of mixed failures were recorded in this group.

Multilink Primer when applied in combination with CPC achieved low bond strength values. This simplified self-etching adhesive contains a high concentration of hydrophilic monomers. The presence of water in the solvent may affect the coupling between the adhesive and the light-cured composite. Moreover, a recent investigation revealed that the high concentration of acidic monomers in the adhesive systems negatively affects the polymerization rate of light-curing composites.

Multilink Primer achieved the best results when it was used with ML cement. The technique of adhesive application on dry dentin, the maintenance of the smear plug within the tubules, and the chemical compatibility of the products produced by the same manufacturer could have contributed to this favorable result. Moreover, polymerization shrinkage stresses that were generated because of the highly unfavorable cavity configuration factor of the post space may be partially compensated by the use of slow-setting self-curing resin cements. This probably accounted for the relatively higher percentage of mixed failure that was seen in this group.

The chemical compatibility between the resinous matrix of the fiber posts and the cement (both containing methacrylate resin), may be an additional factor for the low incidence of adhesive failure along the post/cement interface (11.76%). Moreover, the application of a silane-coupling agent on the post surface also contributed to the strengthening of this interface. The majority of the failures showed cement remnants on the post surface (Figure 2).

On the other hand, when the dual-cured adhesive system (Clearfil Photo Bond) was light-activated and used in combination with the light-curing composite (CPC), most of the specimens failed at the cement-dentin interface [Figure 1(a,b)]. This could be the result of shrinkage stresses that developed from the rapid curing of the composite. The use of a translucent fiber post and the good polymerization rate of the selected light-cured composites probably accounted for the good results achieved in the study.

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