

# Bond Strengths of Fiber Posts to Root Canal Dentin Using Various Resin-Based

Luting Agents

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# บทคัดย่อ

ในปัจจุบัน สารยึดติดเรซิน ที่ใช้ยึดเดือยฟันเสริมเส้นใยกับเนื้อฟันในกลองราก ฟันใด้รับการพัฒนาจนมีความหลากหลายในท้องตลาด ในขณะที่ข้อมูลการใช้งานของแต่ละชนิด ยังมีน้อย ดังนั้นจึงควรให้ความสนใจกับชนิดของสารยึดติดเรซินที่มีผลต่อค่ากำลังแรงยึดระหว่าง เคือยฟันเสริมเส้นใยกับเนื้อฟันในคลองร ากฟัน ซึ่งมีอิทธิพลต่อความสำเร็จของวัสคุบูรณะฟันที่ รักษาคลองรากฟันมาแ<mark>ล้ว</mark>

วัตถุประสงค์ เพื่อศึกษาค่ากำลังแรงยึดระหว่างเดือยฟันเสริมเส้นใยกับเนื้อฟันใน คลองรากฟันเมื่อใช้สารยึดติดเรซินชนิดต่างๆ

วิธีการ ทำการศึกษาในฟันกรามน้อยรากเดียว จำนวน 25 ซี่ ถอนด้วยเหตุผลของ การจัดฟันและเป็นฟันที่มีคลองรากเดียวปราศจากการผุ, วัสดุอุดฟัน, และไม่เคยผ่านการรักษาคลอง รากฟันมาก่อน หุ้มรากฟันด้วยเรซินคอมโพสิทให้ผนังคลองรากฟันส่วนปลายหนาเท่ากับส่วนต้น เตรียมชิ้นทคสอบ โดยตัดฟันส่วนตัวฟันออกเหนือรอยต่อระหว่างเคลื่อบรากฟันและเคลื่อบฟัน 3 มิลลิเมตร ทำการรักษาคลองรากฟันโดยใช้เครื่องมือขยายคลองรากฟันจากเบอร์ 10 ถึงเบอร์ 25 ที่ ความยาวเหนือจุดยอคปลายสุดรากฟัน 0.5 มิลลิเมตร ล้างคลองรากฟันด้วยน้ำยาโซเดียมใฮโปคลอ ไรท์ 2.5 เปอร์เซนต์และซับแห้ง อุดกลองรากฟันแล้วปิดด้วยวัสดุอุดฟันชั่วกราว เตรียมขยาย ช่องว่างสำหรับเดือยฟันให้มีความลึก 9 มิลลิเมตรและเส้นผ่าศูนย์กลาง 1.5 มิลลิเมตรค้วยหัวกรอ ความเร็วต่ำ แบ่งฟันออกเป็น 5 กลุ่มโดยสุ่ม ทำการยึดเดือยฟันเสริมเส้นใยกับเนื้อฟันในคลองราก ฟีนด้วยสารยึดติดเรซิน 5 ชนิดคือ Variolink II, Panavia F 2.0, RelyX Unicem, ParaBond+ParaCore, และ SE Bond X+ParaCore ตามกำแนะนำของบริษัท ทำการเกี่บฟันไว้ 24 ชั่วโมงที่อุณหภูมิ 37 องศาเซลเซียสแล้วจึงนำรากฟันแต่ละรากมาตัดเป็นชิ้นขนาดความหนา 1 มิลลิเมตร จำนวน 6 ชิ้นจากรากฟันส่วนคอฟันถึงส่วนปลายรากฟันโดยกำหนดให้ 2 ชิ้นแรกเป็น ตัวแทนในส่วนคอฟัน, 2 ชิ้นถัดมาเป็นตัวแทนในส่วนกลาง, และ 2 ชิ้นถัดมาเป็นตัวแทนในส่วน ปลายฟัน จะได้จำนวนชิ้นทดสอบเท่ากับ 10 ในแต่ละกลุ่มย่อย นำชิ้นทดสอบที่ได้ไปทดสอบหาก่า กำลังแรงยึดแบบกดด้วยเครื่องทดสอบเอนกประสงค์ด้วยความเร็ว 0.5 มิลลิเมตรต่อนาที ii

หลังจากนั้นนำชิ้นทดสอบไปส่องด้วยกล้องจุลทรรศน์แบบ 3 มิติ (stereoscopic microscope) เพื่อดู ตำแหน่งของการแตกหัก

ผลการศึกษา RelyX Unicem ให้ค่ากำลังแรงยึดแบบกดเฉลี่ยสูงสุดในทุกระดับ พบว่าทุกกลุ่มมีค่ากำลังแรงยึดที่ระดับรากฟันส่วนคอฟันไม่แตกต่างกันอย่างมีนัยสำคัญทางสถิติ (p>0.05) ที่รากฟันส่วนกลาง ค่ากำลังแรงยึดแบบกดเฉลี่ยของกลุ่ม RelyX Unicem และ ParaBond+ParaCore สูงกว่ากลุ่มอื่นมากกว่าสองเท่า (p<0.05) ที่รากฟันส่วนปลาย Variolink II มี ค่ากำลังแรงขึดแบบกดเฉลี่ยเพียง 2.38±1.80 MPa ซึ่งต่ำกว่ากลุ่มอื่นอย่างมีนัยสำคัญทางสถิติ (p<0.05) ในขณะที่ RelyX Unicem ให้ค่ากำลังแรงยึดแบบกดเฉลี่ยสูงถึง 15.80±8.67 MPa เมื่อ เปรียบเทียบค่ากำลังแรงยึดแบบกดเฉลี่ยของสารยึดติดเรซินแต่ละชนิดในตำแหน่งต่างๆ พบว่ พบว่า RelyX Unicem <sup>"</sup>ไม่มีความแตกซ่างของค่ำกักลังแรงขึดแบบกดในแต่ละตำแหน่ง (p>0.05) ในขณะที่ สารยึดติดเรซินชนิดอื่นมีกวามแตกต่างกันของก่ากำลังแรงยึดแบบกดเฉลี่ยในแต่ละตำแหน่ โดย ความแตกต่างขึ้นอยู่กับชนิดของสารยึดติดเรซิน การวิเคราะห์ความล้มเหลวพบว่าชิ้นทดสอบส่วน ใหญ่แตกที่ตำแหน่งระหว่างสารยึดติดเรซินและผิวคลองรากฟัน

สรุป ชนิดของสารยึดติดเรซินและตำแหน่งช่องว่างสำหรับเดือยฟันมีอิทธิพลต่อ ค่ากำลังแรงขี้ดแบบกด โดยสารขึดติดเรซิน RelyX Unicem ให้้ค่ากำลังแรงขึดแบบกดสูงที่สุด ความ แตกต่างกันของค่ากำลังแรงยึดระหว่างเนื้อฟันภายในคลองรากฟันส่วนคอฟัน, ส่วนกลาง และส่วน ปลายฟันพบได้ในทุกกลุ่มการทดลอง ยกเว้นในกลุ่ม RelyX Unicem

Thesis Title Bond Strengths of Fiber Posts to Root Canal Dentin Using Various Resin-Based Luting Agents Author Miss Wiyada Thongbai-on Major Program Oral Health Sciences Academic Year 2009

#### Abstract

 Nowadays, fiber posts have been increasingly used for restorations of endodontically treated teeth. Various resin-based luting agents have been developed for fiber posts cementation. The bond strength of fiber post to root canal dentin obtained from the recent resin-based luting systems is limited. Therefore, it is important to measure regional bond strengths of fiber post-bonded teeth to determine the most favorable bonding technique for fiber post cementation.

Objective: The purpose of this study was to evaluate the regional push-out bond strengths of fiber posts to root canal dentin using various resin-based luting agents.

Materials and methods: Twenty-five single-rooted premolars extracted for orthodontic reasons were used in this study. Each tooth was examined radiographically to have a single root canal, a closed apex and showing no evidence of a caries lesion or restoration or previous root canal treatment. A light-cure composite resin was placed over all the root surfaces to create a uniform thickness of a wall. The teeth were decoronated perpendicularly to the long axis at 3 mm above the cement-enamel junction level. Each root canal was endodontically instrumented from no.10 to no.25 at a working length of 0.5 mm from the apex. After each instrumentation, the root canal was flushed with 2 ml of 2.5% sodium hypochlorite and dried with adsorbent paper points. The coronal access was filled with a temporary filling material after being obturated with a gutta percha main cone. A post space was prepared to the depth of 9 mm with a diameter of 1.5 mm with low speed dowel drills. The roots were divided into 5 groups according to the resin-based luting agents used: Variolink II, Panavia F 2.0, RelyX Unicem, ParaBond+ParaCore, and SE Bond X+ ParaCore. Fiber posts were luted into the post spaces in accordance with the manufacturers' instructions. After 24-hours storage at  $37 °C$  room temperature, each root was serially sliced into six 1-mm-thick slabs to harvest six slabs v

from the cement-enamel junction towards the apex. Each of the two slabs was considered to represent the coronal, middle and apical portion of the post-space, respectively. Thus, each luting agent provided 30 test specimens, consisting of 10 specimens from each post space region. Pushout tests were performed using a universal testing machine at a crosshead speed of 0.5 mm/min until failure. Failure mode was observed using a stereoscopic microscope.

Results: RelyX Unicem provided the highest bond strength value in every region. At the coronal region, there were no significant differences in bond strengths among all luting agent groups (p>0.05). At the middle region, RelyX Unicem and ParaBond+ParaCore significantly exhibited twofold bond strengths higher than that of the others  $(p<0.05)$ . At the apical region, the bond strength of Variolink II was  $2.38\pm1.80$  MPa, which was significantly lower than the other groups ( $p<0.05$ ), while RelyX Unicem provided the highest bond strength of 15.80±8.67 MPa. Regarding the regional difference, only the bond strengths of RelyX Unicem were not significantly different among 3 post space regions  $(p>0.05)$ . In the other groups, it was found that the significant regional differences in bond strengths varied on the type of luting agents. Analysis of failure modes revealed that most failures occurred at the interface between dentin and resin-based luting agent.

Conclusions: The push-out bond strengths were significantly affected by the type of resin-based luting agents and the root canal regions. RelyX Unicem had higher bond strengths when compared with other materials. The regional differences in bond strengths were presented in all tested groups except RelyX Unicem.

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Wiyada Thongbai-on

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# Chapter 1

### Introduction

The practice of fixed prosthodontics has been changed with the introduction of innovative techniques and materials. Dental cements are present examples of these changes that amend the selection guidance of the material for cementation. Clinicians now have many choices of cementation medium to use with fixed restorations, such as water-based luting agent groups, for example, zinc phosphate, zinc polycarboxylate, glass ionomer; an oil-based luting agent group, for example, reinforced zinc oxide-eugenol and a resin-based luting agent group, for example, self- or dual-cure resin composite used with or without an adhesive. Most of the current resin cements are variations of filled BIS-GMA resin and methacrylates. Unfilled resins have been used for cementation since the  $1950s$ .<sup>1</sup> These early products were unsuccessful because of their high polymerization shrinkage and poor biocompatibility even though they had very low solubility. Therefore, small amounts of fillers were added to the resins to improve their properties and decrease polymerization shrinkage. Some resin cements are available with adhesive properties as they are capable of bonding chemically to dentin.<sup>2</sup> Bonding is usually achieved with hydroxyethyl methacrylate (HEMA), or 4-methacryloxyethyl-trimellitic anhydride (4-META), or an organophosphonate such as  $10$ -methacryloyloxydecamethylene phosphoric acid (MDP).<sup>3</sup> These developments remarkably increased the interest in using resin cements for crowns and conventional fixed prostheses cementation. Moreover, with the high demand for esthetic dentistry these days requiring all-ceramic and laboratory-processed composite restorations (instead of traditional amalgam fillings), these kinds of restorations always require highest strength from the bonding procedures that resin cements can provide. In addition to bonding restorative materials to coronal enamel and dentin, resin-based luting agents are also used in the root canal for the bonding of endodontic posts. Since the ability of resin cement to retain endodontic posts influences the prognosis of the restoration, $\frac{4}{3}$  several studies proved a significant increase in the retention of prefabricated posts luted with resin-based cements compared with those luted with conventional cements.<sup>5-7</sup>

Apart from marketing data supplied by the manufacturer, information about new self-adhesive resin cements for luting fiber posts to root canal dentin is still scarce. 1

Additionally, the method of using a dual-cure resin composite core material combined with contemporary adhesive systems for fiber post cementation was recommended by the manufacturer due to the simplicity and homogeneity of using the same material for the post and core placement. The bond strength obtained from this method has never been studied in comparison with the method of using resin cement. Therefore, it has become important to measure regional bond strengths along the length of the canal of human teeth to assess the bond strengths of various systems of resin-based luting agents to endodontically relevant dentin. Owing to any method of enhancing the bond strengths of fiber posts to root canal dentin, the advantage would be beneficial in leading to a higher survival rate of fixed restorations.

### Review of Literatures

#### Restorations of endodontically treated teeth

Endodontically treated teeth that have severe coronal damage are mostly restored with posts and cores before placement of the final restorations. The primary function of a coronoradicular post is to provide retention for a core, which replaces lost coronal tooth structure and retains the final restoration.<sup>8-10</sup> The post is cemented into the root canal and the core is retained by an apical extension.<sup>11, 12</sup> Various types of posts were described and recommended in various literatures. The conventional treatment of severely damaged and endodontically treated teeth is the cast post and core. Prefabricated metal posts, in combination with composite cores, were described as treatment alternatives in the  $1970s^{13, 14}$  and since the mid-1980s, nonmetal posts [fiber-reinforced composite (FRC) and ceramic posts] have been introduced.

The amount of tooth structure that remains after endodontic treatment and post space preparation plays an important role in the survival of restored endodontically treated teeth.<sup>15-17</sup> Factors such as extended caries, trauma to an immature tooth, pulpal pathology or iatrogenic causes may result in a flared root canal with thin remaining dentinal walls. A thin peripheral dentinal wall might be prone to fracture if a metal cast post and core is used, due to its high stiffness and the wedging effect that occurs within the root structure.<sup>18, 19</sup> Firstly, the resin post was introduced and primarily used in order to reduce the incidence of root fracture in compromised teeth. Fiber-reinforced composite posts have been widely used to restore endodontically treated teeth to this day. Some studies<sup>20-22</sup> reported that the fracture resistance of teeth restored with a custom cast post was lower than that of teeth restored with fiber posts. Saupe et al.,<sup>20</sup> Rosentritt et al.<sup>21</sup> and Akkayan and Gulmez<sup>22</sup> demonstrated that the fracture strength of the teeth restored with fiber- reinforced posts was higher than that of metal cast posts. Even though Isidor et al.,<sup>23</sup> Martinez-Insua et al.,<sup>24</sup> and Sirimai et al.<sup>25</sup> reported that the teeth restored with metal cast posts and cores had a higher fracture strength than that of fiber post and cores, failure patterns of teeth restored with metal cast posts were characterized by root fractures.<sup>26-29</sup> On the other hand, the teeth restored with fiber posts frequently showed more favorable failure patterns, which can be restorable through post fractures and loss of retention.<sup>30</sup> The favorable failure mode was a result from the modulus of elasticity of the FRC post that was close to that of dentin, which subsequently decreased the incidence of root fractures.<sup>24</sup>

Finite element (FE) analysis of post restored teeth have frequently been performed<sup>31-33</sup> to reveal the factor that affects the failure modes. Results of FE analyses are expressed as stresses distributed in the structures under investigation. Deformations and stresses in any point of the model can be evaluated, and the stressed areas can be visualized.<sup>33</sup> In FE analysis, an FRC post demonstrated a response similar to a natural tooth except that stress concentration at the cervical margin might be due to microleakage or gaps at the restoration margins. $33$ 

Post decementation was the most frequent failure of endodontically treated teeth restored with post-and-core systems,<sup>34</sup> while vertical root fracture was the most serious type of failure.<sup>35, 36</sup> Adhesively luted posts improved retention when compared to conventionally cemented posts.<sup>5, 37, 38</sup> Therefore, they might reduce the incidence of decementation. FE analysis additionally demonstrated that the bonding of posts was a major factor in reducing stresses inside the root canal. Consequently, FRC posts which when luted with adhesive bonding into the root canal, should be a major factor in preventing root fractures.<sup>31</sup>

Furthermore, many retrospective clinical studies  $\frac{8, 12, 39, 40}{8}$  reported that fiber posts had high clinical success rates. Ferrari et al.<sup>39</sup> annotated that the fiber posts had a higher success rate than custom cast posts after 4 years of service. Subsequently routine recalls were accomplished, Composiposts (carbon fiber post) showed 95% success, which was higher than 84% for custom cast posts.

In addition to having a slightly higher flexibility under load distributed stresses to root canal dentin in a more favorable manner than metal posts, the other advantages of using fiber posts are esthetics and easier removal processes when the teeth need to be retreated. Fabrication of a metal cast post is time consuming and involves additional laboratory cost. An extra dental appointment is required to fit the cast post and core and to take an impression for the subsequent crown. On the other hand, a fiber post can be cemented immediately after canal preparation, and the core can be promptly built, shaped, and an impression taken within a single visit.

#### Fiber posts

There are currently three types of prefabricated fiber posts according to the fiber used; carbon fiber posts (CFP), quartz fiber posts, and glass fiber posts. The fiber content usually ranges from about 35-65%, with a higher fiber content post typically having greater strength and stiffness. The fibers are bound with resin such as epoxy or polyester resins. A carbon fiber post was the first generation of fiber-based posts introduced by Duret *et al.* in 1990,<sup>41</sup> which was made of stretched aligned carbon fibers embedded in an epoxy-resin matrix<sup>42, 43</sup> such as Composinost (RTD, Meylan, France), C-post (Bisco, Inc., Schaumburg, USA), Tech 2000 (Isasan, Rome, Italy), and CARBOPOST (Carbotech, Ganges, France). A retrospective study of clinical service after three years showed that the carbon fiber post was a viable alternative to traditional cast metal posts and core or metal prefabricated posts.<sup>11</sup> The extensive disadvantages of the carbon fiber post were its dark color, which was a major obstacle to the esthetic restoration of an endodontically treated tooth, and its radiolucent appearance which renders invisible in a radiograph. Esthetic requirements are fulfilled with the developments of tooth-colored quartz and glass fiber posts which are available in wide varieties of characteristics in the market. Glass fiber posts were later introduced in  $1992^{44}$  to overcome a color problem in carbon fiber posts by replacing them with unidirectional glass fibers embedded in a resin matrix that strengthens the post without compromising the modulus of elasticity<sup>45</sup> such as ParaPost Fiber White (Coltène/Whaledent, Altstätten, Switzerland), FRC Postec Plus (Ivoclar Vivadent, Schaan, Liechtenstein), FibreKor (Pentron clinical technologies, LLC, USA), and REFORPOST (Angelus, Londrina, PR, BR). Quartz fiber posts were also introduced to use as an esthetic fiber post such as D.T.Light-Post (RTD, St Egreve, France), Luscent Anchor (Dentatus, NY, USA), and Aestheti-Plus<sup>™</sup> (RTD, St Egreve, France). Both glass and quartz fiber posts are made in white, either in translucent or opaque form. The translucent post will allow for light transmission. These tooth-colored fiber posts in the market are mostly fabricated in glass fiber post types. Although Galhano<sup>46</sup> reported that quartz fiber posts were stronger than glass fiber posts, several dentists still used glass fiber posts due to its reasonable price. Therefore, the composite posts containing glass fiber are more available in the dental market.

#### Failures of fiber post restoration

At present, fiber posts bonded to root canal dentin via resin cements are increasingly employed for the restoration of endodontically treated teeth. Although an in vitro study showed that the occurrence of root fractures was rare with the use of fiber posts  $2^2$ , most clinical studies indicated that fiber post restorations might fail via dislodging of the bonded posts or "decementation". <sup>11, 39, 47-49</sup> The high bond strength at post-resin cement and resin cement-dentin interfaces is important when the fiber post is used because of their flexibility that allows the post to slightly bend during function. The flexible post decreases the load transferred to the root structure and prevents root fracture. Several studies showed that failures occurred at the resin cement-dentin interface more often than at the post-resin cement interface.<sup>48, 50-55</sup> Therefore, the bonding to root canal dentin should be circumspectly performed.

#### Luting agents for fiber posts

Various luting agents and corresponding adhesive systems have been used for bonding endodontic posts to root canal dentin. High bond strengths can be achieved with resin cements compared to a glass ionomer and zinc phosphate cement.<sup>5-7, 56</sup> Several types of resin cement systems are currently available in the market. According to the bonding substrate modification, there are three major categories of the resin cement commonly used, as in Table 1. The first one, the "*Etch-and-Rinse*" system, utilizes phosphoric acid etching that completely dissolves the smear layer and creates a zone of demineralized dentin. After rinsing the acid conditioners, hydrophobic resins with or without adhesive are applied to the demineralized dentin to achieve micromechanical retention (i.e. Variolink  $II^{\mathbb{R}}$ , Calibra<sup>TM</sup>, Nexus<sup>®</sup>). Conversely, the second system, the "Self-etch" resin cements, utilizes adhesive primer containing high concentration of acidic resin monomers to simultaneously demineralize and infiltrate the smear

layer-covered dentin prior to resin luting (i.e. Panavia  $21^{\circ}$ , Panavia  $F^{\circ}$ , Panavia F  $2.0^{\circ}$ , Multilink® ). A further reduction in working steps has been accomplished with the recent introduction of "**Self-adhesive**" resin cements (i.e. RelyX<sup>TM</sup> Unicem, Maxcem<sup>TM</sup>, BisCem<sup>TM</sup>, MonoCem<sup>TM</sup>) which do not require any pre-treatment of tooth substrates. As a result of various types of resin cement being commercially available, many authors attempted to compare their bonding strengths when they were used to bond fiber posts to root canal dentin. Some studies demonstrated higher bond strengths achieved by using an etch-and-rinse system compared to selfetch and self-adhesive resin cements.<sup>57, 58</sup> On the other hand, Bitter<sup>52</sup> reported that self-adhesive resin cement demonstrated a higher bond strength than the others. It should be noted that the bond strength data collected from previous studies was divergent and could have come from different experimental conditions.

| Categories      | Examples of products | Manufacturer                             |  |  |
|-----------------|----------------------|--|--|--|
| Etch-and-Rinse  | Variolink II         | Ivoclar Vivadent, Schaan, Liechtenstein  |  |  |
| system          |                      |  |  |  |
|                 | Calibra              | Dentsply Caulk, Milford, DE, USA         |  |  |
|                 | <b>Nexus</b>         | Sybron-Kerr, Orange, CA, USA             |  |  |
| Self-etch resin | Panavia 21           | Kuraray Medical Inc., Tokyo, Japan       |  |  |
| system          |                      |  |  |  |
|                 | Panavia F            | Kuraray Medical Inc., Tokyo, Japan       |  |  |
|                 | Panavia F 2.0        | Kuraray Medical Inc., Tokyo, Japan       |  |  |
|                 | Multilink            | Ivoclar Vivadent, Schaan, Liechtenstein  |  |  |
| Self-adhesive   | RelyX Unicem         | 3M ESPE, St.Paul, MN, USA                |  |  |
| resin system    |                      |  |  |  |
|                 | Maxcem               | Kerr, Sybron Dental Specialties, Orange, |  |  |
|                 |                      | Calif                                    |  |  |
|                 | <b>BisCem</b>        | Bisco Inc., Schaumburg, IL, USA          |  |  |
|                 | MonoCem              | Shofu Dental, San Marcos, CA, USA        |  |  |

Table 1. Three major categories of the resin cement.

Besides the utilization of resin cement for post cementation, the use of an adhesive system with dual-cure resin composites was found to be another effective procedure for bonding endodontic posts to the root canal wall.<sup>59, 60</sup> Dentists always assume that composites couple well to dentin adhesives. In fact, the incompatibility between these two materials has already been clarified. $61$  The decrease in the microtensile bond strength of chemical-cure composites to dentin was reported as being inversely proportional to the acidity of simplifiedstep self-etch adhesives.<sup>62</sup> Cheong<sup>63</sup> demonstrated superior bond strength when using two-step self-etch adhesives with dual-cure resin composite compared with one-step self-etch adhesives. This incompatibility between the self-etch adhesive and chemical- or dual-cure resin composite was caused by an adverse acid-base reaction of the uncured acidic resin monomers with the tertiary amines used in the self-cure initiator systems. Consequently, the amines became inactive as reducing agents, resulting in poor polymerization.<sup>61</sup> Recently, some researchers suggested using photo-cure bonding agents with sufficient light curing for bonding to radicular dentin in order to avoid the incompatibility problem.<sup>59</sup> However, studies that compared the effectiveness of these bonding techniques to the resin cement luting procedures in root canal dentin are limited.

#### Difficulties in bonding to root canal

There are many factors that can weaken adhesion to the root canal wall compared to coronal dentin bonding. Reduced visibility for bonding inside the root canal is one major problem. It is somewhat difficult for an operator to control bonding procedures and apply a consistent layer of any dentin conditioner or adhesive. Another interfering factor is the unfavorable geometry of the root canal system for bonding.<sup>64, 65</sup> Configuration factor or C-factor, the ratio of the bonded to unbonded surface, is often used as a quantitative measure of the geometry of the cavity preparation for bonding. The greater the percentage of unbonded surfaces, the less stress is generated from polymerization contraction. Unbonded surfaces allow plastic deformation, or flow, within the resin mass during polymerization. Virtually every dentin wall has an opposing wall and there are minimal unbonded surfaces. Any ratios greater than 3:1 are considered unfavorable for bonding.<sup>66</sup> In root canal systems, the ratio might be  $100:1$ <sup>65</sup>. A very restricted system such as bonding in a root canal would be a detrimental factor to deteriorate the bonding quality of fiber posts. Variations in the root structure such as accessory root canals, areas of resorption, embedded and free pulp stones, and varying amounts of irregular secondary dentin may influence bonding to root canal dentin. $67$  Moreover, in endodontically treated teeth, the obturation and post space preparation as shown by SEM analysis revealed large areas covered by smear layers, debris, and sealer/gutta-percha remnants on canal walls along the post space.<sup>68</sup> Regarding regional difference in the bond strength, some studies reported higher bond strengths in the apical third than in other parts of the root canal<sup>52, 69</sup>, whereas other studies found a reduction in bond strengths in the apical region of the root canal.<sup>58, 70, 71</sup> Evaluation of dentin morphology in root canals in terms of tubule orientation and density revealed a higher density in the cervical compared with the middle and apical parts of the root canal.<sup>72</sup> However, the tubular density was not found to be a factor that affected the bond

strength when the self-etch adhesives were used.<sup>73</sup>

It can be realized from the above problems that many factors can weaken the bonding of fiber posts to a root canal wall. Accordingly, the clinician should therefore circumspectly select the proper bonding technique in order to obtain the optimal bonding performance to the radicular dentin.

#### Bond strength testings

Several microtensile bond strength studies were performed to evaluate the bonding efficiency of resin cements in root canals. The microtensile bond strength technique (Fig.1) was first introduced by Sano.<sup>74</sup> This technique was credited with the potential to more closely reflect the true interfacial bond strength, the ability to measure adhesion to small surfaces, the capacity to assess local variations over the bonding substrate, and the convenience of obtaining multiple specimens from a single tooth.<sup>75</sup> Microtensile testing methods were originally developed for testing the ultimate tensile strength of dental tissue, but later applied to bond strength measurements on dentin and enamel surface. However, reliability of microtensile tests to assess the bonding of fiber posts to intact dowel spaces was challenged recently<sup>76</sup> with the frequent observation of premature bond failures during specimen preparation. The "thin slice" push-out test (Fig.2) is emerging as a practical tool for evaluating the interfacial shear behavior of the attachment of fiber posts to intact root canals. <sup>57</sup> The resistance to dislocation of fiber posts bonded to intact root canals with resin- or glass-ionomer-based cements may be considered as a net sum of micromechanical interlocking, chemical bonding and sliding friction. For this reason, push-out test results have been more successfully employed as indicators of the interfacial

strengths between fiber posts or filled luting resin and the root canal wall.<sup>50, 77, 78</sup> Similar to the microtensile bond test, an additional advantage of using the "thin slice" push-out test is that multiple specimens may be retrieved from one bonded root canal.



Fig.1 A diagram of the microtentile test (adapted from Goracci *et al.*<sup>76</sup>). A post-cemented root was longtitudinally cut  $(1)$  into 1 mm-thick slabs  $(2)$  that were serially sectioned into sticks  $(3-4)$ . Individual sticks were mounted to special grips for a tensile bonding test  $(5)$ and the tensile bond strength was calculated as the maximum load at failure.



Fig.2 A diagram of the thin slice push-out test (adapted from Goracci *et al.*<sup>76</sup>). A post-cemented root was sectioned into 1 mm-thick slabs. On each slice, the post is loaded until the bond failure occurs and the post fragment is extruded from the root slab.

## Objectives of the study

- 1. To evaluate the regional bond strengths of fiber posts bonded to root canal dentin using various resin-based luting agents by means of the thin-slice push-out method.
- 2. To examine the interfaces of root canal dentin bonded with various resinbased luting agents using a scanning electron microscope (SEM).

# Expected outcomes

The results would give scientific data regarding the regional bond strengths of fiber posts in root canals using various resin-based luting agents, and it may help dentists to select the proper luting medium for bonding fiber posts to root canal dentin.

# Null Hypothesis

The type of luting agent and regional factor did not affect the bond strengths of fiber posts to root canal dentin.

# Chapter 2

# Materials and methods

### 1. Scope of study

This study is an *in vitro* study using endodontically treated human lower premolar teeth. The study was performed to investigate the bond strengths of fiber posts bonded to root canal dentin using various resin-based luting agents.

# 2. Materials

- 2.1 Human lower premolar teeth
- 2.2 Prefabricated glass fiber-reinforced composite posts (Parapost Tenax Fiber White® ; Coltène/Whaledent, Altstätten, Switzerland) in Fig.3



 Fig.3 Photographs of prefabricated glass fiber-reinforced composite posts (Parapost Tenax Fiber White®).

### 2.3 Resin-based luting agents (Fig.4)

- 2.3.1 Variolink II®; Ivoclar Vivadent, Schaan, Liechtenstein : Lot. K49612
- 2.3.2 Panavia F 2.0® ; Kuraray Medical Inc., Tokyo, Japan : Lot.51277
	- 2.3.3 RelyX<sup>TM</sup> Unicem; 3M ESPE, MN, USA : Lot.312491
- 2.3.4 ParaCore Automix/ParaBond® ; Coltène/Whaledent, Altstätten, Switzerland : Lot.0142216
- 2.3.5 Clearfil SE Bond  $X^{\mathbb{R}}$ ; Kuraray Medical, Inc., Tokyo, Japan : Lot. 81146





- Fig.4 Photographs of resin based luting agents used in the present study. A. Variolink  $II^{\mathcal{B}}$ , B. Panavia F 2.0<sup>®</sup>, *C*. RelyX<sup>™</sup> Unicem, *D*. ParaCore Automix/ParaBond<sup>®</sup>, and *E*. Clearfil SE Bond  $X^{\circledast}$ .
	- 2.4 Adhesive agents (Scotch bond multipurpose<sup>®</sup>; 3M Co., MN, USA : Lot. 20080320)
	- 2.5 Light cure resin composite (Filtek<sup>TM</sup> Z250; 3M ESPE, St Paul, MN, USA :  $Lot.6JR)$
	- 2.6 Auto-polymerizing acrylic resin (GC Pattern resin® ; GC Corp, Tokyo, Japan : Lot.0809043)
	- 2.7 Gutta percha main cone no.25 (ProTaper Universal Gutta Percha<sup>®</sup>; Dentsply Maillefer, Ballaigues, Switzerland : Lot.865005)
- 2.8 Eugenol free sealer (AH 26<sup>®</sup>; Dentsply DeTrey, Konstanz, Germany : Lot.0804001692)
- 2.9 Temporary filling (Cavit<sup>®</sup>; 3M ESPE AG, Seefeld, Germany : Lot.02061260)
- 2.10 Cyanoacrylate adhesive (Zapit®; Dental Ventures of America Inc, CA, USA: Lot.M04A)
- 2.11 2.5% Sodium hypochlorite
- 2.12 Distilled water

## 3. Equipment

- 3.1 Light curing unit (Elipar trilight<sup>®</sup>; 3M ESPE, Seefeld, Germany)
- 3.2 Endodontic micromotor (X-smart®; Dentsply Maillefer, Ballaigues, Switzerland)
- 3.3 NiTi root canal rotary instrument (ProTaper® ; Dentsply Maillefer, Ballaigues, Switzerland)
- 3.4 Dowel tools (Peeso drills® ; Dentsply Maillefer, Ballaigues, Switzerland)
- 3.5 Dowel tools (Parapost drills® ; Coltène/Whaledent, Altstätten, Switzerland)
- 3.6 Microtip applicators
- 3.7 Cement spatula
- 3.8 Glass lab
- 3.9 Linear Precision Saw (ISOMET 4000; Buehler Ltd., Ilinois, USA)
- 3.10 Stereoscopic microscope (SMZ1500, Nikon, Tokyo, Japan)
- 3.11 Universal testing machine (LRX-Plus; Lloyd Instrument Limited., Hants, UK)
- 3.12 Digital micrometer (Mitutoyo micrometer; Mitutoyo corp., Tokyo, Japan)
- 3.13 Scanning Electron Microscope (model 5800 LV, Tokyo, Japan)
- 3.14 Scanning Electron Microscope (model Quanta 400, FEI Co., Oregon, USA)

## 4. Groups of study

The teeth were randomly allocated into 5 groups with simple random sampling (SRS). Group 1 comprised of the Variolink II, Group 2 comprised of the Panavia F 2.0, Group 3 comprised of the RelyX Unicem, Group 4 comprised of the ParaBond and ParaCore, and Group 5 comprised of the SE Bond X and ParaCore (Table 2) (Fig.5).

Table 2. Groups of study.





Fig.5 A diagram of the sequential procedures in each resin-luting agent group.

### 5. Methodology

#### 5.1 Tooth sample collection

Twenty five lower premolar teeth recently extracted for orthodontic reasons from adolescents were used in this study. Each tooth was examined radiographically (Fig.6) to have a single root canal, a closed apex and no evidence of a caries lesion or restoration or a root canal treatment. Teeth with excessive root curvature of more than 15 degrees were excluded. After extraction, the teeth were cleaned with a blade to remove any calculus or debris tissue, and then stored in 4°C distilled water for no more than 90 days following extraction.



 Fig.6 Radiographic examination of each tooth. *<sup>A</sup>*, a radiograph was taken in the bucco-lingual view. *B*, a radiograph was taken in the mesio-distal view.



Fig.7 A diagram of each tooth after build-up of a composite resin wall.

#### 5.2 Preparation of tooth sample

Phosphoric acid 37% was applied over the root-surface for 15 seconds prior to priming and bonding (Scotch bond multipurpose; 3M Co., MN, USA) in accordance with the manufacturer's instructions. An A 3.5 light-cure composite resin (Filtek<sup>TM</sup> Z250; 3M ESPE, St Paul, MN, USA) was placed over all the etched root surfaces (Fig.7) to create a cylinder shape with thickness of 2 mm in order to eliminate any effect of perimeter curing light that might interfere with the subsequent post bonding process.

The teeth were decoronated perpendicularly to the long axis of the teeth at 3 mm above the cement-enamel junction level (Fig.8) using a diamond blade with a low speed precision saw (ISOMET 4000, Buehler Ltd., Illinois, USA) under copious water cooling.



Fig.8 A photograph of each root after decoronation.



Fig.9 Photographs of an obturated root (*<sup>A</sup>*) and temporary filling (*B*).

Each root canal was endodontically instrumented with a NiTi root canal rotary instrument (ProTaper; Dentsply Maillefer, Ballaigues, Switzerland) from no.10 to no.25 (diameter = 1.2 mm) at a working length of 0.5 mm from the apex by means of an endodontic micromotor (X-smart; Dentsply Maillefer, Ballaigues, Switzerland) operating at 400 rpm according to the manufacturer's instructions. After each instrumentation, the root canal was flushed with 2 ml of 2.5% sodium hypochlorite and dried with adsorbent paper points. Each canal was obturated with a no.25 gutta percha main cone (ProTaper Universal Gutta Percha; Dentsply Maillefer, Ballaigues, Switzerland) and a eugenol free sealer (AH 26; Dentsply DeTrey, Konstanz, Germany) and mixed according to the manufacturer's instructions up to the cement-enamel level (Fig.9.*A*). The access chamber was filled with a temporary filling (Cavit; 3M ESPE AG, Seefeld, Germany) (Fig.9.*B*), and all root canals were stored in distilled water at 37 °C for 24 hours to allow a complete setting of the sealer.

#### 5.3 Post-space preparation

Each root was embedded in polymerizing acrylic resin (GC Pattern resin<sup>®</sup>; GC Corp, Tokyo, Japan) to form a base, and made parallel to the vertical spindle of a surveyor. Each post space was prepared with a dowel tool (Peeso drills; Dentsply Maillefer, Ballaigues, Switzerland) in the handpiece attached to a vertical spindle of a surveyor from no.1 (diameter  $= 0.70$  mm) to no.3 (diameter  $= 1.10$  mm). Then, the canal wall was enlarged with low speed dowel drills (Parapost drills; Coltène/Whaledent, Altstätten, Switzerland) from red (diameter  $= 1.25$  mm) to black (diameter  $= 1.5$  mm) under copious water cooling to achieve a 9-mm depth post space (measured from the cement-enamel junction), following the manufacturer's instructions. Following the preparation, the post spaces were rinsed for one minute with 2.5% sodium hypochlorite. A final irrigation was accomplished with distilled water, and then the post spaces were dried with paper points (Fig.10.*A*). A parallel permanent pen line was marked along the root surface.

All prepared roots were divided into five groups of five specimens each according to the resin-based luting agented used.

> Group 1) Etch-and-rinse system (Variolink II) Group 2) Self-etch system (Panavia F 2.0) Group 3) Self-adhesive system (RelyX Unicem) Group 4) Dual-cure two-step self-etch adhesive + Dual-cure resin core material (ParaBond+ParaCore Automix) Group 5) Photo-cure two-step self-etch adhesive + Dual-cure resin

> > core material (SE Bond X+ParaCore Automix)

#### 5.4 Bonding procedure

The taper part of twenty-five prefabricated glass fiber-reinforced composite posts (Parapost Tenax Fiber White; Coltène/Whaledent, Altstätten, Switzerland) were removed and the post surfaces were wiped with alcohol for cleaning and treated with a silane agent (Monobond-S®; Ivoclar Vivadent, Schaan, Liechstein) over the surface. Then each post was cemented into a prepared root canal with various resin-based luting agents as mentioned (Table 3). After cementing, the post was shortened to flush the coronal portion of the root and then covered by a composite resin (Fig.10.*B*). All roots were stored in water for 24 h at 37 °C

until testing.



Fig.10 Photographs of a root after post-space preparation (*<sup>A</sup>*) and after bonding (*B*).

| Materials                 | Primer/Etchant               | Adhesive                 | Luting resin compositon          | Application                                     |  |
|---------------------------|------------------------------|--------------------------|----------------------------------|---|--|
|                           | (Product:Composition)        | (Product:Composition)    |                                  |   |  |
| Group.1                   | Total etch : phosphoric acid | Syntac adhesive : PEGDA, | Base: Bis-GMA, UDMA,             | -Apply Total Etch (phosphoric acid gel 37%)     |  |
| Variolink <sup>®</sup> II | gel 37%                      | Glutaraldehyde, Water    | TEGDMA, Barium glass filler,     | to the prepared cavity in the root canal for 15 |  |
|                           |                              |                          | Silanated Ytterbium trifluoride, | s using a microbrush tip.                       |  |
|                           | Syntac primer : Maleic acid, |                          | Mixed oxides, Silanated Ba-Al-   | -Clean and rinse with water using an            |  |
|                           | TEGDMA, Acetone, Water       |                          | fluoro-silicate glass, Catalysts | endodontic syringe, then dry canal with paper   |  |
|                           |                              |                          | and stabilizers, Pigments        | points avoiding excessive drying.               |  |
|                           |                              |                          |                                  | -Apply Syntac primer to the cavity and brush    |  |
|                           |                              |                          | Catalyst: Bis-GMA, UDMA,         | for 15 s. Excess is removed with paper points   |  |
|                           |                              |                          | TEGDMA, Barium glass filler,     | and dried with gentle air flow.                 |  |
|                           |                              |                          | Silanated Ytterbium trifluoride, | -Apply Syntac adhesive to the cavity, leave     |  |
|                           |                              |                          | Mixed oxides, Silanated Ba-Al-   | 10 s. Excess is removed with paper points and   |  |
|                           |                              |                          | fluoro-silicate glass, Catalysts | dried with gentle air flow.                     |  |
|                           |                              |                          | and stabilizers, Pigments        | -Mix base and catalyst components of            |  |
|                           |                              |                          |                                  | Variolink II in equal proportion.               |  |
|                           |                              |                          |                                  | -Carry mixture into root canal.                 |  |
|                           |                              |                          |                                  | -Insert the post and light-cure for 40 s.       |  |

Table 3. Composition and application method of materials used in this study.







# Table 3. (Continued)

Table 3. (Continued)

| Group.5           | SE-Primer: Silanated silica. | $SE$ -Bond $X: 2$ -hydroxyethyl | ParaCore: Methacrylates. | -Apply SE-Primer for 20 s with a microtip               |
|-------------------|------------------------------|---------------------------------|--------------------------|---|
| ParaCore Automix/ | Bis-GMA, 2-hydroxyethyl      | methacrylate, Hydrophilic       | Fluoride, Barium glass,  | applicator and gently air dry                           |
| SE Bond X         | methacrylate, Hydrophilic    | dimethacrylate, 10-MDP,         | Amorphous silica         | -Apply SE-Bond X  |
|                   | dimethacrylate, 10-MDP       | N, N-Diethanol p-toluidine.     |                          | -Gently air dry and light-cure the bonding for          |
|                   | Toluidine, Camphorquinone    | Camphorquinone (2,3-            |                          | 20s.  |
|                   |                              | bornanedione), Water            |                          | -Apply ParaCore <sup>TM</sup> automix directly from the |
|                   |                              |                                 |                          | cartridge into the post space.                          |
|                   |                              |                                 |                          | -Insert the post and light-cure the cement for          |
|                   |                              |                                 |                          | 20 s.   |

#### 5.5 Push-out testing

Each specimen was attached to the arm of the low speed diamond saw (ISOMET 4000, Buehler Ltd., Illinois, USA) and vertical cuts were made perpendicular to the bonded interface under water-cooling to harvest six slabs, of approximately 1.00 mm thickness, from the cement-enamel junction towards the apex. Each pair of two slabs was considered respectively to represent the coronal, middle and apical portion of the post-space (Fig.11). Thus, each study group of five roots provided 30 test specimens, consisting of 10 specimens from each of the three different post space regions (Fig.5). The exact thickness of each section was measured using a digital micrometer to an accuracy of  $\pm$  0.01 mm (Fig.12.*A*). Before push-out testing, the specimens were examined under a stereoscopic microscope (SMZ1500, Nikon, Tokyo, Japan) to clarify the void, and also, cement thickness was measured at 4 points; at the mid-surface of the buccal, lingual, distal, and mesial part of the root.



Fig.11 A diagram of each pair of two slabs in the root, which represented the coronal, middle and apical portions of the post-space.



Fig.12 Photographs of the digital micrometer showing the 1-mm thick measured specimen (*A*) and the examples of cut slabs (*B*).

Push-out tests were performed using two customized stainless steel pushout devices; a loading plunger and support. The section was centered over the space between the supports by marking lines intersecting at the center of each section. A cyanoacrylate adhesive (Zapit; Dental Ventures of America Inc, CA, USA) was applied on the composite resin portion of the section for fixing it to the supporting device during testing.

A compressive load was applied to the slice via a universal testing machine (LRX-Plus, Lloyd Instrument Limited., Hants, UK) equipped with a 1.3 mm diameter cylindrical plunger. Loading was performed at a crosshead speed of 0.5 mm/min until failure (Fig.13), as manifested by the complete extrusion of the post segment from the root slice. This was further confirmed by the appearance of a sharp drop along the load/time curve recorded by the testing machine.



Fig.13 A diagram of the push-out test using a universal testing machine.

#### 5.6 Statistical analysis

The bond strength (MPa) was computed by dividing the load at the time of debonding by the area (A) of the bonded interface. The latter was calculated using the formula A=2¶rh where *r* represented the post radius and *h* represents the thickness of the slice in mm. The data were analyzed using a two-way ANOVA followed by Post-hoc multiple comparisons. Levene's test was used to test the homogeneity of variances. Tukey's HSD was used to compare the bond strength in each group when the homogeneity of variances was presented, whereas Dunnett's T3 was used when there was no homogeneity of variances. The average cement thickness of each slab was calculated and the cement thickness values in each experimental group were compared using One-way ANOVA. All statistics analyses were performed at the 95% level of confidence.

#### 5.7 Failure analysis and SEM observation

Fractured slices were carefully removed and observed under a stereoscopic microscope (SMZ1500, Nikon, Tokyo, Japan) at 20x to categorize the type of failure as follows: (a) Cohesive fracture of root dentin; (b) Adhesive fracture at root dentin-luting material interface; (c) Cohesive fracture of luting material; (d) Adhesive fracture at luting material-post interface; (e) Cohesive fracture of post; or  $(f)$  Mixed failure, a combination of two of the aforementioned types of adhesive failure. Each type of failure was classified when that failure pattern occupied over 80% of all the area. Resin-dentin interdiffusion zones (hybrid layer) and resin tag formations were examined with a scanning electron microscope (SEM) (model 5800 LV, Tokyo, Japan). A separate specimen preparation protocol was used for the SEM observations, and a specimen from each group was similarly prepared as described for the push-out test specimens.<sup>79</sup> The root sections were cut parallel to the long axis of the tooth in a mesial-distal direction using the same low-speed saw under water cooling after storage in distilled water for 24 hours. The resulting 2 dentin-adhesive interface specimens for each group were prepared for SEM by using the following technique: one section of each tooth was gently decalcified with 32% phosphoric acid for 30 seconds, rinsed with distilled water, and subsequently deproteinized by immersion in a 2% NaOCl solution for 120 seconds to evaluate the resin-dentin interdiffusion zone formation.<sup>79</sup> After rinsing with water and air drying, the specimens were mounted on brass tablets and coated with gold sputter. Cervical, middle, and apical regions of each specimen were examined with an SEM (model 5800 LV, Tokyo, Japan) and an SEM (model Quanta 400, FEI Co., Oregon, USA) at magnification, x1000.

# Chapter 3

## Results

## Regional push-out bond strength

Two-way ANOVA revealed that the bond strengths were significantly affected by the resin-based luting agents  $(p<0.0001)$  and the post space regions  $(p<0.0001)$  (Table 4). There was no significant interaction between the resin-based luting agents and the post space regions (p=0.474).





Independent variables: resin-based luting agent, post space region; dependent variable: bond strength; df, Degree of freedom.

|         | Variolink II | Panavia F 2.0 | RelyX Unicem ParaBond+  |          | SE Bond X+ |
|---------|--------------|---------------|---|----------|------------|
|         |              |               |   | ParaCore | ParaCore   |
| Coronal |              |               | $10.92 \pm 7.52$ <sup>A</sup> 12.56 $\pm$ 4.64 <sup>A</sup> 18.72 $\pm$ 8.67 <sup>A</sup> 17.44 $\pm$ 6.00 <sup>A</sup> 15.49 $\pm$ 4.35 <sup>A</sup> |          |            |
| Middle  |              |               | 6.59±2.62 <sup>M</sup> 6.24±2.50 <sup>M</sup> 17.05±6.30 <sup>N</sup> 14.21±5.69 <sup>N</sup> 6.81±2.20 <sup>M</sup>                                  |          |            |
| Apical  |              |               | 2.38±1.80 <sup>x</sup> 5.13±1.90 <sup>x</sup> 15.80±8.67 <sup>z</sup> 10.29±6.84 <sup>x,z</sup> 6.36±1.84 <sup>x,z</sup>                              |          |            |

Table 5. Regional push-out bond strength values (mean±SD) in MPa.

\*Mean values with the same letter in each row indicated no significant difference at p>0.05. \*Mean values connected with a vertical line in each column indicated no significant difference at p>0.05.



Fig.14 A histogram to demonstrate the means and standard deviation values of the push-out bond strengths calculated for all resin-based luting agents at coronal, middle, and apical post space regions.

The bond strengths and standard deviations (SDs) at the coronal, middle, and apical regions for each group were shown in Table 5 and Fig.14. Results revealed that RelyX Unicem provided the highest bond strength in every region, but there were no significant differences in bond strengths among all luting agents at the coronal region ( $p>0.05$ ). At the middle region, RelyX Unicem  $[17.05 \pm 6.30$  MPa] exhibited comparable bond strength to ParaBond+ParaCore  $[14.21 \pm 5.69 \text{ MPa}]$  (p>0.05), and they were significantly higher than those of other cements. There were no significant differences in bond strengths among the groups of Variolink II, Panavia F 2.0, and SE Bond X+ParaCore in this region  $(p>0.05)$ . At the apical region, the bond strength of RelyX Unicem [15.80±8.67 MPa] was not statistically different from ParaBond+ParaCore [10.29± 6.84 MPa] (p=0.701) and SE Bond X+ParaCore [6.36±1.84 MPa] (p=0.060), whereas the bond strength of Panavia F 2.0 [5.13 $\pm$ 1.90 MPa] was not significantly different from ParaBond+ParaCore and SE Bond X+ParaCore as well ( $p>0.05$ ). The lowest bond strength was obtained from Variolink II  $[2.38\pm1.80$  MPa], which was significantly lower than the other groups  $(p<0.05)$ .

Regarding the regional differences, the bond strengths decreased in the deeper regions. However, significant differences were found to be various depending on the type of luting agent. The results demonstrated that only the bond strengths of RelyX Unicem were not statistically significantly different among the three regions  $(p>0.05)$ . For Variolink II, the lowest bond strength was obtained at the apical region, whereas the bond strengths at the coronal and middle regions were not significantly different ( $p>0.05$ ). For Panavia F 2.0 and SE Bond X+ParaCore, the highest bond strengths were shown at the coronal region whereas the bond strengths of the middle and apical regions were similar, and they were about two times lower than those at the coronal region. For ParaBond+ParaCore, significant difference in bond strength was indicated only between coronal and apical regions.

|         | Variolink II                 | Panavia F 2.0                  | RelyX Unicem                   | ParaBond+                    | SE Bond X+                   |
|---------|------------------------------|--------------------------------|--------------------------------|------------------------------|------------------------------|
|         |                              |                                |                                | ParaCore                     | ParaCore                     |
| Coronal | $0.10 \pm 0.04$ <sup>A</sup> | $0.16 \pm 0.08$ <sup>A</sup>   | $0.11 \pm 0.03^{\text{A}}$     | $0.16 \pm 0.12^{A}$          | $0.16 \pm 0.12$ <sup>A</sup> |
| Middle  | $0.11 \pm 0.09^{\rm B}$      | $0.09 \pm 0.07^{\rm B}$ D      | $0.08 \pm 0.03^{\mathrm{B}}$   | $0.07 \pm 0.06^{\rm B}$      | $0.10\pm0.08^{\rm B}$        |
| Apical  | $0.08 \pm 0.07^\mathrm{C}$   | $0.07 \pm 0.04$ <sup>C</sup> l | $0.05 \pm 0.01$ <sup>C</sup> l | $0.05 \pm 0.01$ <sup>C</sup> | $0.05 \pm 0.02^{\circ}$      |

Table 6. Cement thickness values (mean±SD) in mm.

\*Mean values with the same letter in each row indicated no significant difference at p>0.05. \*Mean values connected with a vertical line in each column indicated no significant difference at  $p > 0.05$ .

Concerning the defects of cement in filling the post space, the cement layers of all specimens examined under the stereoscopic microscope had no void existence in the substance. One way-ANOVA was used to test the difference in cement thickness. There was no significant difference in cement thickness among tested resin-based luting agent groups (p>0.05). However, the cement thickness was found to be affected by post space regions. Generally, the cement thickness values at the coronal region were higher than those at the middle and apical regions, respectively. The statistically significant differences of cement thickness between coronal and apical regions were indicated for Panavia F 2.0 and RelyX Unicem groups (p<0.05), whereas there were no significant differences in cement thickness among post space regions for other tested resin-based luting agent groups  $(p<0.05)$ . Although there were no statistical differences in cement thickness between coronal and apical regions for ParaBond+ParaCore and SE Bond X+ParaCore groups, the significance was presented at  $p=0.063$  and  $p=0.052$ , respectively.

#### Failure analysis

number  $(\%)$ ).

|               | Specimen number $(\% )$ |                  |                  |                  |          |          |  |
|---------------|-------------------------|------------------|------------------|------------------|----------|----------|--|
| Luting agent  | Adhesive                | Adhesive         | Cohesive         | Cohesive         | Cohesive | Mixed    |  |
|               | dentin/luting           | post/luting      | luting           | dentin           | post     | failure  |  |
| Variolink II  | 27(90.00)               | $\boldsymbol{0}$ | $\boldsymbol{0}$ | $\boldsymbol{0}$ | $\theta$ | 3(10.00) |  |
| Panavia F 2.0 | 27(90.00)               | 1(3.33)          | $\theta$         | $\boldsymbol{0}$ | $\theta$ | 2(6.67)  |  |
| RelyX Unicem  | 29(96.67)               | 1(3.33)          | $\boldsymbol{0}$ | $\boldsymbol{0}$ | $\theta$ | $\theta$ |  |
| ParaBond      | 22(73.33)               | 2(6.67)          | $\boldsymbol{0}$ | $\boldsymbol{0}$ | $\theta$ | 6(20.00) |  |
| $+ParaCore$   |                         |                  |                  |                  |          |          |  |
| SE Bond X     | 27(90.00)               | $\theta$         | $\boldsymbol{0}$ | $\boldsymbol{0}$ | $\theta$ | 3(10.00) |  |
| $+ParaCore$   |                         |                  |                  |                  |          |          |  |

Table 7.Frequency distribution of the specimen numbers in each failure pattern (Specimen

In the assessment of failure modes under a stereoscopic microscope, the results of failure patterns of each luting agent were shown as the frequency distribution of specimen numbers in each type of failure mode (Table 7). It was found that the cohesive failures within dentin or posts were not observed in this study. For all luting resin groups except ParaBond+ParaCore, approximately 90% of specimens failed as an adhesive failure at the interface between the dentin and resin-based luting agent (Fig.15). For this type of failure, the stereo-micrograph showed that the entire luting agent was still attached to the fiber post after push-out testing. There were only a few specimens that failed as an adhesive failure between the post and luting agent. The adhesive failure between the resin-based luting agent and fiber post was seen, as the entire luting agent attached to the dentin after the push-out testing without cement remnants on the post surfaces (Fig.16). For the mixed failure, the failure patterns of the adhesive fracture at the root dentin-luting material interface and the adhesive fracture at the luting material-post interface were observed. The characteristic of the mixed failure was seen as there were fractured lines inside the resin layer and cement remained on both sides of dentin and post surfaces (Fig.17).



Fig.15 A stereo-micrograph showing an adhesive failure between a resin-based luting agent and dentin. The entire luting agent was still attached to the fiber post after push-out testing.



Fig.16 Stereo-micrographs showing an adhesive failure between a resin-based luting agent and a fiber post. The entire luting agent attached to the dentin after push-out testing without cement remnants on the post surface.



Fig.17 A stereo-micrograph showing a mixed failure. After push-out testing, the cement remnants remained on both sides of dentin and post surfaces.

# SEM observation





Fig.18 SEM photomicrographs showing the post-resin-dentin interfaces obtained from 3 luting agents; Variolink  $II$  demonstrated numerous resin tags  $(R)$  and clear hybrid layers  $(H)$ , Panavia F 2.0 demonstrated some short resin tags and consistent hybrid layers, and RelyX Unicem demonstrated no resin tags with unidentified hybrid layers (original magnification x 1,000).



Fig.19 SEM photomicrographs showing the post-resin-dentin interfaces obtained from 2 luting agents; ParaBond+ParaCore demonstrated a few short resin tags and consistent hybrid layers, and **SE Bond X+ParaCore** demonstrated a few short resin tags and consistent hybrid layers (original magnification x 1,000).

In this study, SEM observation was used afterwards to evaluate the quality of hybrid layers and resin tag formations at the interface between fiber posts and luting agents. The results showed that all five resin-based luting agents provided good adaptation to the root canal dentin without any gaps (Fig.18-19). For Variolink II, several intact resin tags were observed with a well-defined hybrid layer, whereas RelyX Unicem presented an unidentified hybrid layer and its resin tag was not found. For the systems utilizing self-etch primer before bonding, Panavia F 2.0, ParaBond+ParaCore, and SE Bond X+ParaCore, the appearances of the interfaces were similar. Only a few short resin tags could be observed. The hybrid layer seemed to be consistent although the resin tags were not formed. Moreover, the surfaces of root canal dentin before bonding were observed in all groups by SEM. The results showed that there was no different among three regions.

# Chapter 4

## **Discussions**

In this in vitro study, investigation of the bond strength of fiber posts to root canal dentin using various resin-based luting agents used a push-out test representing a shear stress at the interfaces between dentin and cement as well as between post and cement $\frac{80}{10}$ , which is comparable to the stresses under clinical conditions. A premature bond failure of the test specimens, which was a sign of unreliability in the bond strength test<sup>76</sup>, was not found in any resin-based luting agent testing groups of this study. The testing results proved that the null hypothesis was rejected. The bond strengths did vary with the type of resin-based luting agent and the post space regions inside the root canal.

The present results showed that RelyX Unicem provided the highest bond strength among five resin-based luting agents in all regions. Additionally, the canal depth had no effect on the bond strength of RelyX Unicem. This reason might be due to the high moisture tolerance behavior and the material having less technique sensitivity. After using distilled water irrigation in the last process of the root canal or endodontic treatment, even though the root canal was carefully dried by using paper point adsorption, some water might remain on the dentin surface especially within the dentinal tubules due to poor visibility and difficulty in water removal. Furthermore, the narrow tubules hold water by surface tension, making it difficult to displace water with bonding agents.<sup>81</sup> RelyX Unicem has high moisture tolerance behavior because it has a typical monomer in the constituent that can react with basic salts and tooth apatite in the tooth structure. The typical monomer contains at least two phosphoric acid groups and a minimum of two double bonded carbon units  $(C=C)$  per molecule which provides the acidity of pH 1 at the beginning, reaching to pH 5 within 5 mins, and up to pH 7 within 24 hours to function simultaneously both in demineralization of the tooth surface and penetration of the cement into the demineralized surface. The monomer reaction is done through the functional groups which were modified by phosphoric acid. Water is consequently formed in this neutralization. This step will increase hydrophilicity, improve adaptation of the luting agent to the tooth structure, and enhance moisture tolerance. Additionally, the methacrylated phosphoric esters contained in RelyX Unicem have a strong chemical interaction with 35 hydroxyapatite.<sup>82</sup> The above mentioned characteristic of RelyX Unicem might cause this cement to provide the highest bond strength in this study. Similarly to a previous study, Bitter *et al.*<sup>52</sup> investigated the push-out bond strength of six luting agents: Panavia F, Multilink, Variolink II, PermaFlo DC, RelyX Unicem, and Clearfil Core. RelyX Unicem also provided significantly higher bond strength than other materials.

Besides the moisture tolerance capability of resin monomers, the characteristics of the dentinal surface may be another factor that affects the bonding quality. In this study, the drilled canals were designed to be slightly larger than the endodontic obturated canals. The remaining root canal sealer and gutta-percha after canal preparation was expected to be entirely removed. The uncontaminated dentin surface would facilitate the bonding of single-step resin cements as RelyX Unicem compared to the dense sealer covered dentin. Some previous studies<sup>57, 58</sup> reported low bond strengths when the root canal dentin was bonded with selfadhesive cements. The features of the dentin after canal preparation might be the reason for the low bond strength in those studies. In the deep and narrow canal, it was difficult to observe the dentin surface before bonding. If some debris remained in the canal, they would weaken the bonding effectiveness, especially for the luting system that had no pretreatment step before luting such as the RelyX Unicem. Therefore, the studies<sup>52, 83</sup> that were conducted to evaluate the bond strength to root canal dentin reported controversial results obtained from each luting system. In this study, the root canal filling and sealer materials were totally removed to control the characteristics of the dentin surface. Moreover, in a real clinical situation, post sizes are always chosen to be slightly larger than the canal sizes. The SEM photomicrograph of the dentin surface after canal drilling was taken to confirm the dentin characteristic before bonding. Dentinal tubules filled with smear plugs were observed in some areas, while some areas covered with smear layers. The resin sealer-filled tubules were rare (Fig.20).



Fig.20 An SEM photomicrograph showing the surface of root canal dentin before bonding. Dentinal tubules filled with smear plugs were observed in some areas, while some areas covered with smear layers. The resin sealer-filled tubules were rare.

The results of the resin cement utilized etch-and-rinse system, Variolink II, and the self-etch system, Panavia F 2.0, showed similar bond strengths at both coronal and middle post space regions, except at the apical post space region where the bond strength of Variolink II was significantly lower than that of Panavia F 2.0 at  $p=0.035$ . The apical bond strength of Variolink II was two times lower than Panavia F 2.0. This might result from the technique sensitivity problem. The application procedures of the etch-and-rinse system, Variolink II, were extremely technique sensitive. After etching and rinsing, moist dentin is required for optimal bond strengths. However, the limitations of accessibility and visibility during bonding procedures were obstacles for moisture control. Moreover, the Variolink II system requires syntac adhesive application before resin luting. The syntac adhesive has no initiator, and it requires an initiator for polymerization from the luting resin. Due to the limitation of light energy, the free radicals, which initiate the polymerization process, were possibly too small for both syntac adhesive and resin luting. Therefore, the adhesive might not be adequately cured, resulting in poor bond strengths, especially at the apical region. The self-etch system, Panavia F 2.0, had less technique sensitivity compared to the etch-and-rinse system. ED primer in the Panavia F 2.0 system was applied and followed by air blowing to remove water from the primer, then the luting resin was inserted. The bond strength obtained from Panavia F 2.0 was not statistically different from the other luting agents at the coronal region. However, the remaining water content might deteriorate the adhesion at the deeper portion because it is fairly difficult to evaporate the water content in the primer by the air blowing method inside the deep and narrow

canal. In addition, incompatibility between acidic ED Primer and dual cure luting resin might be another reason for low bond strength of Panavia F 2.0 at the middle and apical of the canal.<sup>61</sup>

For the systems utilizing contemporary adhesives combined with core material, ParaBond+ParaCore provided high bond strength values similar to RelyX Unicem for all regions. The ParaBond system requires non-rinse conditioner application prior to a dual-cure adhesive. This system utilizes the self-etch primer for dentin treatment the same as Panavia F 2.0. However, the dual-cure adhesive is additionally applied before ParaCore luting resin. The low viscosity adhesive might increase the wettability to the dentin surface and improve the adaptation at the resin-dentin interface. Moreover, water and ethanol are used as the solvents in this adhesive. The evaporation of the co-solvent, water-ethanol, is easier than that of water alone. The remaining solvent might be less than when using Panavia F 2.0. The bond strength was therefore quite high, and only slightly decreased at the deeper region. On the contrary, the SE Bond X group requires a light-cure adhesive after self-etch primer application. This more hydrophobic light-cure adhesive was firstly introduced to apply in the root canal to reduce the problem of incompatibility between acidic primer and dual-cure adhesive. It was reported in a previous study<sup>84</sup> that SE Bond was effective for bonding to root canal dentin when it was sufficiently photo-irradiated. However, different light curing machines and different operators might create different results between the present study and previous studies. Even though the photoirradiation time of SE Bond X in this study was extended from 10 seconds to 20 seconds for assuring the sufficiency of polymerization of the adhesive, the bond strength values in the deeper part were still low. If strong light irradiation could not be performed, the dual-cure adhesive such as ParaBond might be more suitable for bonding in the root canal.

Regarding the regional difference of the bond strength, the results indicated statistical differences among three regions for all luting agents except the RelyX Unicem group. Some differences presented between coronal and middle regions, whereas some presented between middle and apical regions. The factors that affected the regional difference might be the degree of conversion of the resin and technique sensitivity. The highest bond strengths were generally found at the coronal post space regions, which might be the result of sufficient light energy that irradiated from the coronal end. Moreover, the coronal portion has good accessibility for bonding procedures. In contrast to the middle and apical post space regions, light penetration is limited compared to the coronal portion. The resin might not be completely cured resulting in

a declination of the bond strengths. Studies by Lui<sup>85</sup> and Takahashi *et al.*<sup>86</sup> also supported that the regional bond strengths were affected by photo energy, which decreased due to the depth of the post space. However, the results in this study showed that RelyX Unicem had only a small decrease in bond strength in the apical post space region. The self-adhesive cement system, RelyX Unicem, has the least technique sensitivity; it does not require any pretreatment of the tooth substrate. Once the cement was mixed, application was accomplished through a single clinical step. The resin can simultaneously etch and penetrate into demineralized dentin. Without the primer or adhesive application, any residue would not be left due to mistakes induced by technique sensitivity. Therefore, the bond strengths were not significantly affected by the post space regions for the RelyX Unicem group.

SEM observation was used to evaluate the quality and quantity of the hybrid layer and resin tag formation in each resin-based luting agent. All five resin-based luting agents presented good adaptation to the root canal wall (Fig.18-19). RelyX Unicem provided the highest bond strength, although the hybrid layer was unidentified and there was no resin tag formation. On the other hand, Variolink II resulted in the lowest bond strength while many intact resin tags were formed. A recently published study by Bitter *et al.*<sup>87</sup> found a similar interface appearance in a RelyX Unicem group. They observed a low number of resin tags, while the Variolink II presented a higher number of them. In addition, the results of bond strengths of that previous study<sup>87</sup> and the present investigation were also similar. It seems that characteristics shown in SEM photomicrographs might not be able to represent the quantitative bond strengths. As in this study and the previous study,  $\frac{87}{10}$  the amount of resin tags was not related to the quantitative bond strength. The predominant factor that created the high bond strength of RelyX Unicem, might be the chemical interaction between the adhesive in resin cement and tooth structure. This was also supported by the previous study of Gerth *et al.*<sup>82</sup> that RelyX Unicem had an intense chemical reaction between the carboxylic groups of polyalkenoic acid and calcium of hydroxyapatite (Hap) by the methacrylated phosphoric esters.

For failure analysis, most of the resin-based luting agents had more than 90% of specimens fail as adhesive failures between dentin and cement, while isolated cohesive failure inside the cement and adhesive failures between post and cement, were rare. It indicated that the bond strengths to root canal dentin were weaker than the bond strengths between post and cement. Many in vivo and in vitro studies<sup>48, 52-55</sup> have also reported that adhesive failures

predominantly occurred between dentin and cement. The superior bond strength at the post and luting resin surface in this study was established by treatment of the post surface with a silane coupling agent before bonding the post into the root canal. The capability of a silane coupling agent in wetting the post surface and creating chemical interaction to the silica-contained component of the fiber post would enhance the bonding between the post surface and luting resin.<sup>88</sup> Likewise, several studies<sup>89-92</sup> supported that a silane agent can improve the bond strengths between the fiber post and a resin-based luting agent. The failures therefore took place at the dentin surface.

It can be noticed that the standard deviation (SD) values were considerably large for most of the experimental groups. It was more difficult to detect the statistical difference when SD was high, although the bond strength values appeared different. For example, the bond strengths at the coronal region of the RelyX Unicem and Variolink II were 18.72 and 10.92 MPa respectively, but they were not statistically different at  $p=0.069$ . The bond strength studies often encountered high SD values and they finally seemed to be the nature of the bond strength test, especially to the tooth structure. The difference in individual teeth is difficult to control even though the criteria for root selection in this experiment was specified, such as the selection for a consistent shape of the canals. The cylindrical drill, which is larger than canal size, was also used to make a steady canal shape for every tooth. An exact round canal shape from the coronal to apical end could still not be obtained. There were some areas of canals that had irregular shape (Fig.21). Inconsistency in canal shape might be one of the reasons for wide-ranging SD. Regarding the control of the cement thickness, the post was inserted parallel to the long axis of the canal, which was drilled using a surveyor tool. However, the cement thickness at the coronal region was higher than that of the apical region in every luting agent. Statistical analysis revealed the differences of cement thickness between coronal and apical regions for Panavia F 2.0 and RelyX Unicem (p<0.05). Although the statistically significant differences in cement thickness between coronal and apical regions for ParaBond+ParaCore and SE Bond X+ParaCore were not found at  $p=0.063$  and  $p=0.052$ , respectively, the coronal cement thickness of them were more than twice higher than those of the apical cement thickness. The root canals always have a taper shape toward the apex. It is easier to remove the coronal root canal filling materials with a thin layer of superficial root canal dentin. However, a thicker dentin had to be removed to obtain consistent canal size from the coronal to apical. Inevitably, the coronal post space was loosely

fitted to the drill, whereas the apical post space was tightly fit. The difference in cement thickness might cause different degrees of volumetric shrinkage around the post between coronal and apical regions. A study of Perez *et al.*<sup>93</sup> proved that the bond strengths of resin cements of fiber posts to root canal dentin were not affected by the cement thickness. They reported that the bond strength of a fiber post to the root dentin luting with  $87.4\pm49$  µm-thick resin was not different from the group luted with 316.7±58 µm-thick resin. In the present results, the bond strength at the coronal region of the RelyX Unicem was the same as the apical region, even though the cement thickness values of these two regions were significantly different. However, the resin cements used in the study of Perez *et al.*<sup>93</sup> and this study were different. The effect of cement thickness to the bond strength in the present study needed to be further clarified.



Fig.21 A stereo-micrograph showing the irregular shape in a canal.

Owing to the test set-up, the bonding process in this study was performed using extracted teeth and prepared under a laboratory environment. Under real clinical conditions, the bond strengths of resin-based luting agents to root canal dentin might be different because there are many factors involved with bonding. This study could only evaluate the initial bond strengths. The results might be useful for primary selection of a luting agent for bonding. Since there are many resin-based luting agents available in the market, while the data of bond strengths supplied by the manufacturer about new materials and methods are still scarce. The following further study should be performed for more information; 1) a long term laboratory study to evaluate the durability of the bond strengths or the bond strengths after the teeth received cyclic loading, and 2) a randomized prospective clinical trial to investigate the survival rate of different types of fiber-posts cemented with various types of luting cement.

# Chapter 5

# **Conclusions**

The following conclusions may be drawn from the present study:

- 1. Push-out bond strengths were affected by the type of resin-based luting agents and the post space region inside the root canal.
- 2. RelyX Unicem provided the highest mean push-out bond strengths compared with other materials in every region.
- 3. The regional differences in bond strength were presented for all tested groups except the RelyX Unicem group.
- 4. In this present study, the failure modes were mostly found at the interface between dentin and resin-based luting agents.

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Appendix



1. Table showing the significant values of post-hoc test comparing the bond strengths among

resin-based luting agents.

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2. Table showing the significant values of post-hoc test comparing the bond strengths among three post space regions.



. Table showing the significant values of post-hoc test comparing the cement thickness

among resin-based luting agents.



4. Table showing the significant values of post-hoc test comparing the cement thickness

among three post space regions.

# **Vitae**



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# **Educational Attainment**



# **List of Publication and Proceeding**

**Poster Presentation** 

Thongbai-on W, Smithmaitrie J, Buranadham S, editors. Bond strengths of fiber posts to root canal dentin using various resin-based luting agents. Proceedings of the 19<sup>th</sup> Thaksin University Annual Conference; 2009, Sep 24-25, Songkhla, Thailand.