



รายงานวิจัยฉบับสมบูรณ์

โครงการ

การยึดติดของเดือยฟันเสริมเส้นใยในคลองรากฟัน

Adhesion of fiber post to root canal dentin

luted with dual-cure resin composite

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โครงการ การยืดติดของเดือยฟันเสริมเส้นใยในคลองรากฟัน

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สำนักงานคณะกรรมการอุดมศึกษา และสำนักงานกองทุนสนับสนุนการวิจัย
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กิตติกรรมประกาศ

งานวิจัยนี้สำเร็จลงได้เป็นอย่างดีโดยได้รับการสนับสนุนทุนวิจัยจาก สำนักงานกองทุนสนับสนุนการวิจัยและสำนักงานคณะกรรมการการอุดมศึกษา ซึ่งเป็นหน่วยงานที่สนับสนุนการวิจัยในหลายสาขา และผลการวิจัยก่อให้เกิดประโยชน์แก่ประเทศมากมาย ผู้วิจัยขอขอบพระคุณคณะผู้บริหารและเจ้าหน้าที่ของ สกอ. และ สกว.ทุกท่าน ที่ได้ให้การสนับสนุนและช่วยเหลือในการทำงานวิจัยนี้

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นอกเหนือจากคณะทำงานวิจัย คณะอาจารย์ผู้ร่วมงานในคณะทันตแพทยศาสตร์ มหาวิทยาลัยสงขลานครินทร์ และเจ้าหน้าที่ต่างๆที่ได้ให้การช่วยเหลือ มีส่วนอย่างมากที่ทำให้งานวิจัยนี้สำเร็จได้เป็นอย่างดี ขอกราบขอบพระคุณ คุณพ่อ คุณแม่ ครู อาจารย์ ที่ได้อบรมสั่งสอน ให้การศึกษาจนผู้วิจัยมีวันนี้ได้ ขอขอบคุณสามีที่เปิดโอกาสให้ได้ทำงานวิจัยได้เต็มที่ และให้คำแนะนำที่ดีในการทำงาน ขอขอบคุณลูกๆ ที่เป็นกำลังใจให้ผู้วิจัยทำงานได้โดยมีรู้สึกเหนื่อย

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Project Summary

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ชื่อโครงการ: การยึดติดของเดือยฟันเสริมเส้นใยในคลองรากฟัน

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ความแข็งแรงของพันธะและการวิเคราะห์ความล้มเหลวของการใช้เดือยฟันเสริมเส้นใยใน คลองรากฟัน

บทคัดย่อ 1

งานวิจัยนี้ทำเพื่อศึกษาค่าความแข็งแรงของพันธะระหว่างเดือยฟันเสริมเส้นใยและคลองรากฟัน โดยยึดด้วย เรซินคอมโพสิตชนิดบ่มตัวด้วยแสงร่วมกับปฏิกิริยาทางเคมี การทดลองทำโดยนำฟันกรามน้อยรากฟันเดี่ยวจำนวน 12 ซี่ ตัดให้เหลือแต่ส่วนรากฟัน แล้วใช้หัวกรอสำหรับทำเดือยฟันเตรียมโพรงรากฟันลึก 8 มิลลิเมตร เตรียมผิวคลองรากฟันโดยใช้สารยึดติดชนิด Clearfil SE Bond ทาที่ผิวคลองรากฟัน ฉายแสง 20 วินาที นำเดือยฟันเสริมเส้นใย 4 ชนิดคือ Snowlight FibreKor D.T.Light-Post และ GC Fiber Post ชนิดละ 3 อันมาทาผิวเดือยฟันด้วยสารไซเลนและยึดในคลองรากฟันโดยใช้เรซินคอมโพสิตชนิดบ่มสองทาง หลังจากเก็บขึ้นตัวอย่างไว้ 24 ชั่วโมง นำมาตัดตามลำดับความลึกจากด้านบนโพรงฟัน ให้ได้ชิ้นตัวอย่างสำหรับทดสอบค่าความแข็งแรงของพันธะระดับไมโครจำนวน 8 ชิ้น ต่อเดือยฟันหนึ่งอัน โดยแบ่งเป็นค่าความแข็งแรงของพันธะด้านตัวฟันและปลายฟันอย่างละ 4 ชิ้น นำข้อมูลที่ได้มาทำการวิเคราะห์โดยใช้วิธีทางสถิติ พบว่าปัจจัยเรื่องตำแหน่งคือตัวฟันและปลายฟันไม่มีผลกระทบต่อค่าความแข็งแรงของพันธะ เดือยฟันเสริมเส้นใยชนิด FibreKor ให้ค่าความแข็งแรงของพันธะสูงสุด ในการวิเคราะห์ความล้มเหลวของการใช้เดือยฟันเสริมเส้นใยในคลองรากฟันด้วยกล้องจุลทรรศน์อิเล็กตรอน แบบส่องกราดพบว่า เดือยฟันเสริมเส้นใยชนิด FibreKor และ D.T.Light-Post มีการแตกระหว่างผิวเดือยฟันและเรซินคอมโพสิตที่ใช้ยึด ในขณะที่เดือยฟันเสริมเส้นใยชนิด Snowlight และ GC Fiber Post มีการแตกเกิดขึ้นภายในตัวของเดือยฟันเอง

คำสำคัญ: ความแข็งแรงของพันธะ เดือยฟันเสริมเส้นใย คลองรากฟัน

Regional bond strengths and failure analysis of fiber post bonded to root canal

Abstract 1

This study evaluated the regional bond strengths of fiber posts to root canal dentin luted with dual-cure resin composite. Twelve extracted human premolars were decoronated and post spaces prepared to a depth of 8 mm. The root canal dentin was treated with Clearfil SE Bond and light-cured for 20 seconds. Three of the following fiber posts: Snowlight, FibreKor, D.T. Light-Post, and GC Fiber Post were surface-treated with a mixture of Porcelain Bond Activator and Photobond and then luted into the post spaces with Clearfil DC Core Automix and light-cured for 60 seconds. After 24 hour water storage, each specimen was serially sliced into 8, 0.6 x 0.6 mm-thick beams for the microtensile bond strength (μ TBS) test. Failure modes were observed using SEM. μ TBS data were divided into coronal and apical regions and statistically analyzed. The highest bond strengths were obtained from FibreKor posts. Regional factor had no effect on the bond strength. FibreKor and D.T. Light-Post specimens mostly failed at the post-resin composite interface, whereas Snowlight and GC Post cohesively failed within the post. Bond strengths of fiber post to root canal dentin could be affected by characteristics of the fiber post and its bonding quality. Failure patterns of fiber post bonded root canal dentin were dependent upon the post system.

Keywords: Microtensile bond strength, Fiber post, Root canal dentin

ผลของวิธีการฉายแสงต่อการยึดติดของสารยึดติดระบบเซลฟ์เอทซ์ชั้นตอนเดียวบ่มตัวด้วยแสงร่วมกับปฏิกิริยาทางเคมี

บทคัดย่อ 2

งานวิจัยนี้ศึกษาผลของความเข้มแสงและระยะเวลาการฉายแสง ต่อค่าความแข็งแรงของพันธะเฉพาะตำแหน่งของ Clearfil DC Bond ซึ่งเป็นสารยึดติดระบบเซลฟ์เอทซ์ชั้นตอนเดียวบ่มตัวด้วยแสงร่วมกับปฏิกิริยาทางเคมี ต่อเนื้อฟันในคลองรากฟัน ทำโดยนำฟันกรามน้อยรากเดียวจำนวน 18 ซี่ ตัดให้เหลือแต่ส่วนรากฟัน แล้วใช้หัวกรอสำหรับทำเดือยฟันเตรียมโพรงรากฟันลึก 8 มิลลิเมตร เตรียมผิวคลองรากฟันโดยใช้สารยึดติดชนิด Clearfil DC Bond ทาที่ผิวคลองรากฟัน ฉายแสงเป็นเวลา 10 20 และ 30 วินาที ด้วยเครื่องฉายแสงแบบฮาโลเจนที่มีความเข้มแสงต่างกัน คือ Optilux 501 มีความเข้มแสงวัดได้ 830 มิลลิวัตต์ต่อตารางเซนติเมตร และ Hyperlightel มีความเข้มแสงวัดได้ 1350 มิลลิวัตต์ต่อตารางเซนติเมตร ปิดคลองรากฟันด้วยเรซินคอมโพสิตชนิดบ่มตัวด้วยแสงร่วมกับปฏิกิริยาทางเคมี หลังจากเก็บชิ้นตัวอย่างไว้ 24 ชั่วโมง นำรากฟันมาตัดตามลำดับความลึกจากด้านบนโพรงฟัน ให้ได้ชิ้นตัวอย่างสำหรับทดสอบค่าความแข็งแรงของพันธะระดับไมโครจำนวน 8 ชิ้นต่อรากฟันหนึ่งซี่ โดยแบ่งเป็นค่าความแข็งแรงของพันธะด้านตัวฟันและปลายฟันอย่างละ 4 ชิ้น นำข้อมูลที่ได้มาทำการวิเคราะห์โดยใช้วิธีทางสถิติ พบว่าที่ตำแหน่งรากฟันส่วนต้น ไม่มีความแตกต่างกันของค่าความแข็งแรงของพันธะระหว่างกลุ่มทดลอง ในขณะที่รากฟันส่วนปลาย ค่าความแข็งแรงของพันธะเพิ่มขึ้นเมื่อฉายแสงนานขึ้นเป็นเวลา 30 วินาทีสำหรับเครื่องฉายแสง Optilux 501 และ 20 และ 30 วินาที สำหรับ Hyperlightel นอกจากนี้ผลการทดลองยังพบว่าไม่มีความแตกต่างของค่าความแข็งแรงของพันธะระหว่างส่วนต้นและส่วนปลายคลองรากฟันเมื่อเพิ่มระยะเวลาการฉายแสงให้นานขึ้น

คำสำคัญ: สารยึดติดระบบเซลฟ์เอทซ์ ความแข็งแรงของพันธะ การฉายแสง

Effect of photocuring strategy on bonding of dual-cure one-step self-etch adhesive to root canal dentin

Abstract 2

This study evaluated the effect of light-power density and light-exposure time on regional bond strengths of Clearfil DC Bond to root canal dentin. Post spaces were prepared in extracted premolars. Root canal dentin was treated with a dual-cure bonding system, Clearfil DC Bond, and light-cured for 10, 20 or 30 seconds using two halogen light-curing units: Optilux 501 (830 mW/cm^2) and Hyperlightel (1350 mW/cm^2). All post spaces were filled with a dual-cure resin composite. Microtensile bond strengths (μTBS) at the coronal and apical regions were measured after 24-hour storage. The μTBS at the coronal region was similar among all groups. However, the bond strength at the apical region improved when extending the curing time to 30 seconds with the Optilux 501 and 20 or 30 seconds with the Hyperlightel. Moreover, the significant differences in μTBS between coronal and apical regions disappeared with prolonged curing times.

Keywords: Microtensile bond strength, Dual-cure Self-etch Adhesive, Root canal dentin,

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หน้าสรุปโครงการ (Executive Summary)

1. ชื่อโครงการ

(ภาษาไทย) การยึดติดของเดือยฟันเสริมเส้นใยในคลองรากฟัน

(ภาษาอังกฤษ) Adhesion of fiber post to root canal dentin luted with dual-cure resin composite

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3. สาขาวิชาที่ทำการวิจัย Dental Materials (Adhesion)



5. ระยะเวลาดำเนินงาน 2 ปี

6. ความสำคัญและที่มาของปัญหา (IMPORTANCE AND MOTIVATION OF THE RESEARCH)

Recently, esthetic non-metal fiber posts have been increasingly used to restore endodontically treated teeth because their modulus of elasticity are comparable to dentin resulting in a reduction of root fractures. However, failure of fiber post restored teeth still occurred through decementation between the fiber post-resin and/or the resin-root dentin interfaces.

Our previous researches found that application of a silane coupling bonding agent to the post surface was effective in achieving optimal bond strength between dual-cure composite resin and silica-based fiber posts, and application of a photo-cure adhesive resin with sufficient photo exposure time was most favorable method to treat the root canal

dentin surface. In those studies, the adhesions of resin composite to post surface and resin composite to root canal dentin were separately evaluated. However, when fiber post is luted into root canal, two interfaces; post/resin composite and resin composite/root canal dentin are created. It is still uncertain that which interface actually is a problem of the fiber post restoration, and there is very limited study on this issue. Additionally, when fiber post is bonded to root canal dentin, C-factor enormously increase. The restriction of free surface which is able to reduce the shrinkage stress may affect the bond strength as well. The weakest part of fiber post bonded teeth needed to be indicated in order to improve the success rate of endodontically treated teeth with fiber post restoration.

In another issue, the development of single step all-in-one adhesive resulted in the increase of their using for bonding dental hard tissue primarily due to the simple clinical procedure. However, all-in-one adhesive combines the three functional compositions, etching, priming and bonding, into one and contains ionic resin monomers with acidic phosphate or carboxylic functional groups, hydrophobic monomers, water and an organic solvent. As a result, these adhesives have high hydrophilicity. Some researchers have indicated that single-step all-in-one adhesives behave as semi-permeable membranes that permit diffusion of water even after polymerization. Using all-in-one adhesive in a complex cavity such as a deep and narrow post space within root canal dentin possibly generate more unfavorable effect because of the limitation of the bonding access, and clinicians are not able to work with clear visibility within the root canal. Moreover, the solvent in all-in-one adhesive, which should be totally removed for the optimal bond strength, may not be completely evaporated because of the depth of the root canal. Therefore, it is important to investigate the efficiency of using all-in-one adhesive for bonding to root canal dentin.

7. วัตถุประสงค์ (OBJECTIVES OF THE RESEARCH)

- 1) To evaluate the regional microtensile bond strengths of four kinds of fiber posts to root canal dentin luted with dual-cure resin composites.
- 2) To observe the failure characteristics in order to determine the poor quality part of fiber post bonded root canal
- 3) To evaluate the effect of the light-curing unit, which provides different power densities, and the exposure time on regional bond strengths of dual-cure one-step self-etch adhesive to root canal dentin bonding.

8. ระเบียบวิธีวิจัย (Methodology)

The experiments were separated in two parts

Part 1 Regional bond strengths and failure analysis of fiber post bonded to root canal

Extracted human premolars were decoronated and post spaces prepared to a depth of 8 mm. The root canal dentin was treated with Clearfil SE Bond and light-cured for 20 seconds. Three of the following fiber posts: Snowlight, FibreKor, D.T. Light-Post, and GC Fiber Post were cut to a length of 12 mm and surface-treated with a mixture of Porcelain Bond Activator and Photobond. The posts were then luted into the prepared post spaces with Clearfil DC Core Automix and light-cured for 60 seconds. After 24 hour water storage, each specimen was serially sliced into 8, 0.6 x 0.6 mm-thick beams for the microtensile bond strength (μ TBS) test. Failure modes were observed using scanning electron microscope (SEM). Bond strength data were divided into coronal and apical regions and statistically analyzed.

Part 2 Effect of Photo-curing Strategy on Bonding of Dual-cure One-step Self-etch Adhesive to Root Canal Dentin

Extracted premolars were decoronated at the cemento-enamel junction, and post spaces were prepared to a depth of 8 mm and a diameter of 1.5 mm in the roots. A dual-cure bonding system; Clearfil DC Bond was applied to the root canal dentin in post space and then light-cured for 10, 20 or 30 seconds using two light-curing units; Optilux 501 and Hyperlightel. All post spaces are then filled with a dual-cure resin composite (Clearfil DC Core automix) and light-cured for 60 second. After 24 hour storage, each specimen was serially sliced into 8, 0.6 x 0.6 mm- thick beams for microtensile bond test. . Failure modes were observed using SEM. The bond strength data were divided into coronal and apical regions and statistically analyzed.

9. RESEARCH PLAN

Phase	0.5 Year	1 Year	1.5 Year	2 Year
Design experimental procedures for experiment 1, collect materials, and perform a pilot study				
Perform the experiment1, collect data, and statistically analysis				
Design experimental procedures for experiment 2, collect materials, and perform a pilot study				
Perform the experiment 2, collect data, and statistically analysis				
Write reports/manuscripts				

10. Outputs:

1. Aksornmuang J, Nakajima M, Foxton RM, Panyayong W, Tagami J. Regional bond strengths and failure analysis of fiber posts bonded to root canal dentin. Operative Dentistry, 2008; 33(6): 636-643.
2. Aksornmuang J, Nakajima M, Foxton RM, Panyayong W, Tagami J. Effect of photo curing strategy on bonding of dual-cure one-step self-etch adhesive to root canal dentin. Dental Materials Journal,2009; 28(2): 133-141.

CHAPTER 1 INTRODUCTION

Recently, esthetic non-metal fiber posts have been increasingly used to restore endodontically treated teeth because their moduli of elasticity are comparable to dentin, producing a stress field similar to that of a natural tooth and resulting in a reduction of root fractures (1-5). Contemporary adhesive systems with dual-cure composite resin core materials are currently used for luting fiber posts into root canals especially in a wide or flared canals because resin composite has a modulus of elasticity close to dentin and fiber posts, and higher than that of resin cement (6). In addition, the clinician can simply use the same composite material for post placement and core build-up.

Even though the incidence of root fractures in endodontically treated teeth has reduced when fiber posts have been used, failures of fiber post-restored teeth still occur mostly through decementation between the fiber post-resin and/or the resin-root dentin interfaces (7-10). Polymerization shrinkage and contraction stress of the luting resin composite may be one cause of decementation if sufficient interfacial adhesion could not be obtained. For an attempt to solve debonding problem, our previous studies have been performed to evaluate the bond strength of dual-cure composite resin core materials to fiber post surface and to root canal dentin (11-14). However, those studies separately evaluated the adhesions of resin composite to post surface and resin composite to root canal dentin to determine the most favorable method for bonding each interface. In reality, when a fiber post is luted into the root canal, two different types of the interfaces; post-resin composite and resin composite-root canal dentin are created under the polymerization-stressed condition of the luting resin. Moreover, the cavity configuration factor (C-factor), which represents the ratio of bonded to unbonded surface area, increases enormously for fiber post bonded teeth. It has been reported that the C-factor in endodontic post luted cavities may exceed 200, whereas the C-factor of an intracoronal restoration is in the range of only 1 to 5 (7). The restriction of any free surfaces which might be able to reduce the shrinkage stress would have an additional affect on the adhesion of fiber posts to root canal dentin (15).

Some studies have suggested that problems in adhesion might occur at the resin composite/root canal dentin interface rather than at the fiber post-resin composite interface (9). On the other hand, previous research has also indicated a problem at the post-resin composite interface (16;17). Therefore, it is still uncertain which interface is the weakest part of the fiber post restoration. Vichi and others used scanning electron microscopy (SEM) to evaluate the interfaces created in a fiber post bonded root canal, and they suggested that an absence of voids at the fiber post-resin cement interface indicates a good bond between the post surface

and resin cement (18). However, SEM evaluation alone cannot be correlated to quantitative bond strength data.

Presently, there are two testing methods that mostly used to evaluate the regional bond strength of fiber post bonded teeth; microtensile and push-out test. Microtensile bond strength test enables the measurement transverse force on small bonded area such as the inside of root canal (19), however, occasionally premature failure of specimens arises prior testing when bonding is inferior (20). Thin-slice push-out test has been also useful for evaluating regional retentive ability of fiber post to root canal wall by vertical or shear force loading although frictional resistance may be relevant in push-out test (21). However, this push-out test may not be able to compare the qualities of two resin interfaces of dentin and fiber post with one specimen at the same time. The purpose of this study was to investigate the qualities of two resin interfaces to dentin and fiber post and to identify the weaker part by evaluating the regional microtensile bond strengths of four kinds of fiber posts to root canal dentin luted with dual-cure resin composites. Additionally, the failure characteristics were evaluated to determine the poor quality part of restored teeth with fiber posts. The null hypothesis was that the type of fiber post did not have an effect on adhesion to root canal dentin.

There is another interesting issue regarding the adhesive system used for root canal dentin bonding. As the above mention, dual-cure resin core material in combination with an adhesive system is generally used to bond the fiber post to the root canal wall (6;7;12;22). With a novelty in dentin adhesives, some manufacturers have just developed a dual-cure version of the one-step self-etch adhesive used with resin core materials because it simplifies the application procedures for fiber post luting and core build-up. Since light penetration is limited in the deeper region compared with the upper region of the post cavity, dual-cure adhesive in the deeper region is expected to polymerize by self-cure mode with resin core materials filled into the post cavity.

One-step self-etch adhesive contains acidic resin monomer, water, and organic solvent. A major concern that should be considered is the incompatibility between the acidic monomer of one-step self-etch adhesive and the self- or dual-cure composite resin. Many researchers have reported that an adverse acid-base reaction of acidic resin monomers with the tertiary amines used in the self-cure initiator systems occurred, causing the amines to lose their effectiveness as reducing agents and thus resulting in poor polymerization (23;24). This incompatibility was found to be a barrier to good bonding (25-28). In the deeper region of the post cavity, uncured acidic resin monomer within one-step self-etch adhesive could affect the self-polymerization of dual-cure resin core materials. When completely curing the acidic adhesive, the incompatibility issue and the self-polymerization effect of the resin core materials could be reduced. Therefore, it would be important to irradiate sufficient light energy to the

dual-cure adhesive through the post space in order to minimize the adverse effects of uncured acidic resin monomer within one-step self-etch adhesive.

Additionally, the mechanical properties of dual-cure resin were found to be affected by curing strategies (29;30). Light exposure to the dual-cure adhesive may be important for good bond strength compared with solitary chemical activation because the bond strength was found to be dependent on the mechanical properties of the adhesive resin(31;32). Within the post space, high attenuation of light passing through the canal may jeopardize bonding quality, especially at the bottom part. Previous studies have reported the benefit of using a high intensity light-curing unit to enhance the curing efficiency of the resin, especially in the deep cavity or under tooth-colored restorations (33-35). Likewise, some studies have suggested extending the exposure time to the light- or dual-cure adhesive resin to ensure optimal polymerization in the deep region of the cavity (13;36). Therefore, it can be presumed that using a high intensity curing unit and/or prolonging the light-exposure time to the adhesive resin may improve the adhesion of dual-cure one-step self-etch adhesive to root canal dentin.

From the rationales mentioned above, there were two study designs required to investigate the adhesion of fiber posts to root canal dentin luted with dual-cure resin composite. The purposes of the first study was to investigate the qualities of two resin interfaces to dentin and fiber post and to identify the weaker part by evaluating the regional microtensile bond strengths of four kinds of fiber posts to root canal dentin luted with dual-cure resin composites. Additionally, the failure characteristics were evaluated to determine the poor quality part of restored teeth with fiber posts. The null hypothesis was that the type of fiber post did not have an effect on adhesion to root canal dentin. The second study was done to evaluate the effect of the light-curing unit, which provides different power densities, and the exposure time on regional bond strengths of dual-cure one-step self-etch adhesive to root canal dentin bonding. The null hypothesis was that the light-curing unit, exposure time, and region did not affect the bond strength.

CHAPTER 2 METHODOLOGY

PART 1 Regional bond strengths and failure analysis of fiber post bonded to root canal

Materials used in the study

Material	Manufacturer	Composition
Snowlight	Carbotech, USA	65% volume of zircon- glass fiber, 35% volume of polyester-metacrylate resin matrix
FibreKor	Pentron Clinical Technologies, LLC, USA	30.8% volume of glass fiber, 16.2% volume of filler, 53% volume of resin content
D.T. Light-Post	RTD, Grenoble, France	60 % volume of quartz Fibers, 40 % volume of epoxy resin
GC Fiber Post	GC Corporation, Japan	58% volume of glass fiber, 42% volume of resin matrix
Clearfil SE Bond	Kuraray Medical Inc, Japan	Primer: MDP, HEMA, water, hydrophilic dimethacrylates, photoinitiator, accelerator Bond: MDP, HEMA, hydrophobic dimethacrylates, microfiller, photoinitiator, accelerator
Clearfil Porcelain Bond Activator	Kuraray Medical Inc, Japan	Hydrophobic dimethacrylate, γ - methacryloxy propyltrimethoxy silane (γ -MPS)
Clearfil Photobond	Kuraray Medical Inc, Japan	Catalyst: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, d,l-Camphorquinone, benzoyl peroxide Universal: Ethanol, N,N-Diethanol p-toluidine
DC Core Automix	Kuraray Medical Inc, Japan	Catalyst: Bis-GMA, TEGDMA, silanized glass fillers, silica microfillers, chemical/photoinitiator Universal: TEGDMA, methacrylate monomers, silanized glass fillers, silica microfillers, chemical/photoinitiator

Specimens preparation

Three of the following four fiber posts: Snowlight (Carbotech, Ganges, France), FibreKor (Pentron Clinical Technologies, LLC, USA), D.T. Light-Post (RTD, Grenoble, France), and GC Fiber Post (GC Corporation, Tokyo, Japan), were used in this study. The posts were cut to a length of 12 mm from the upper end by a diamond bur (201, Shofu, Kyoto, Japan) mounted in a high-speed handpiece under water spray (37). The cut posts of Snowlight, FibreKor, and GC Post were straight in shape, while the D.T. Light-Posts were tapered in shape. The surfaces of the posts were then cleaned with alcohol.

Twelve single-rooted human premolar teeth, recently extracted from adolescents for orthodontic reasons and stored frozen, were decoronated at the cemento-enamel junction using a low speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Pulpal tissue was removed using endodontics files and the post spaces were then prepared using Gates-Glidden drills (Matsutani Seisakusho Co., Ltd., Takanezawa, Japan) and FibreKor drills (Pentron Corporation, Wallingford, CT, USA) in a low-speed hand piece under copious water cooling to a depth of 8 mm and a diameter of 1.75 mm. After post space preparation, the root canals were rinsed with distilled water and dried with paper points. Prior to the bonding procedures, the external surfaces of the roots were built up with a composite resin to make grips for testing and to prevent the effect of external light from the curing tip, which can pass through the thin portion of dentine wall to the adhesive resin during the photo curing procedures.

Bonding procedures

Regarding the actual bonding procedures, Clearfil SE Bond Primer/Bond (Kuraray Medical Inc, Tokyo, Japan) was applied to the root canal dentin surface in accordance with the manufacturer's instructions. After application of the adhesive, photo-irradiation (Optilux 501, Demetron, Danbury, USA) was performed from a coronal direction with a prolonged photo-exposure time of 20 seconds (11). The post surface was treated with a silane coupling bonding agent, a mixture of Clearfil Photobond and Clearfil Porcelain Bond Activator, followed by gentle air blowing (11). Dual-cure resin composite, Clearfil DC Core Automix, (Kuraray Medical Inc, Tokyo, Japan) was injected into the post space using an auto-mix cartridge and syringe tip. The treated post was then inserted into the resin-filled root canal, and photo-irradiation was performed for 60 s from the upper end of the post. The specimens were then stored in water at 37 °C for 24 hours.

Microtensile bond strength testing

After 24 hours storage, each bonded specimen was attached to the arm of a low speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) and 8 slabs were serially cut perpendicular to the bonded interface under water cooling. Each slab was then transversely sectioned at the middle part of the post into approximately 0.6×0.6 mm-thick beams. The cross-sectional area of each beam was measured using digital calipers (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan). One of two interfaces of each beam was randomly selected for testing. The ends of the beam and the remaining interface were glued to a testing device in a table-top testing machine (EZ Test, Shimadzu Co., Kyoto, Japan) using cyanoacrylate glue (Zapit, DVA, Anaheim, CA, USA) and subjected to a tensile force at a crosshead speed of 1 mm/min (Figure 2.1). The microtensile bond strength data of the coronal four beams were considered to represent the coronal portion of the post space corresponding to the coronal third of the root canal, and the apical four beams data were considered to represent the apical region corresponding to the middle third of the root canal.

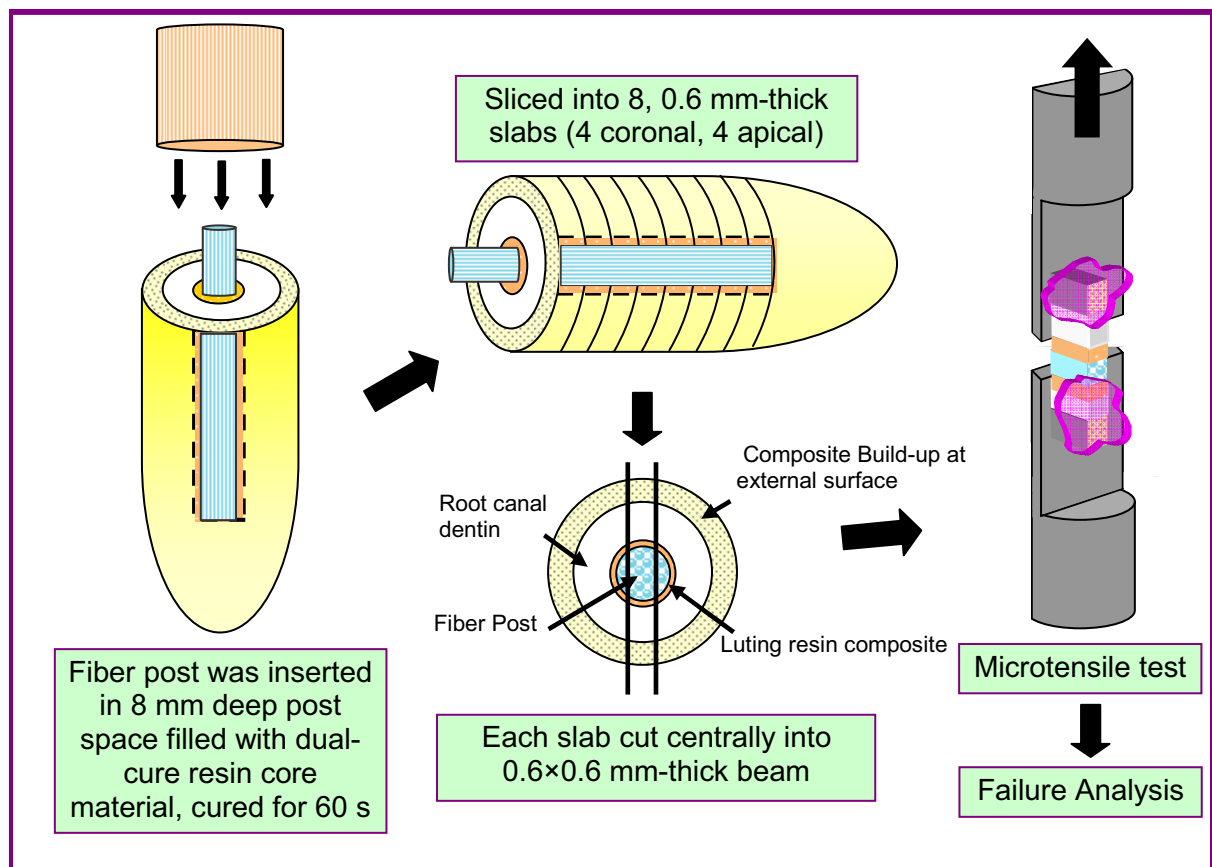


Figure 2.1 Schematic illustration of the bonding and μ TBS test procedures in PART 1

SEM Observation

For failure analysis, both sides of the fractured beams were mounted on brass tablets and gold sputter-coated. The fracture modes were observed using a scanning electron microscope (JSM-5310, JEOL, Tokyo, Japan). The fracture mode was classified into five patterns: cohesive failure within the post, cohesive failure within resin composite, failure at the post/resin composite interface, mixed cohesive failure within resin composite and failure at the post/resin composite interface. Since failure only occurred at the fiber post interface without dentin interface failure, unbonded posts were mounted on brass tablets and gold sputter-coated for observation of the post surface.



Figure 2.2 Fractured beams mounted on brass tablets and gold sputter-coated.

Statistical analysis

The μ TBS data were analyzed using two-way ANOVA to test the affect of post type and region factors on the bond strength. In addition, the interaction between these two factors was tested. Tukey's HSD was used as a pos-hoc test for multiple comparison. All statistic analyses were performed at a 95% level of confidence.

PART 2 Effect of Photo-curing Strategy on Bonding of Dual-cure One-step Self-etch Adhesive to Root Canal Dentin

Materials used in the study

Materials	Manufacturer	Composition
Clearfil DC Bond	Kuraray Medical Inc, Japan	<u>A liquid</u> : MDP, hydrophobic dimethacrylates, HEMA, photoinitiator, chemical catalyst, nanofiller <u>B liquid</u> : water, ethanol, chemical catalyst
DC Core Automix	Kuraray Medical Inc, Japan	<u>Catalyst</u> : Bis-GMA, TEGDMA, silanized glass fillers, silica microfillers, chemical/photoinitiator <u>Universal</u> : TEGDMA, methacrylate monomers, silanized glass fillers, silica microfillers, chemical/photoinitiator

Preparation of bonded specimens

Eighteen single-rooted human premolar teeth, recently extracted from adolescents for orthodontic reasons and stored frozen, were decoronated at the cemento-enamel junction using a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). Pulpal tissue was removed using endodontic files, and post spaces prepared using Gates—Glidden drills (Matsutani Seisakusho Co. Ltd., Takanezawa, Japan) and FiberKor drills (Pentron Corporation, Wallingford, CT, USA) in a low-speed handpiece under copious water cooling to a depth of 8 mm and a diameter of 1.5 mm. After preparation, the post spaces were rinsed with distilled water and dried with paper points. Prior to the bonding procedure, external surfaces of the roots were built up with composite resin (Clearfil DC Core; Kuraray Medical Inc, Tokyo, Japan) to make grips for testing and to prevent the effect of external curing light — which can pass through the thin portion of dentin wall to the bonding agent during irradiation.

A dual-cure one-step self-etch adhesive system, Clearfil DC Bond (Kuraray Medical Inc, Tokyo, Japan) was used according to the manufacturer's instructions for bonding to root canal dentin. Equal amounts of liquid A and B were mixed for five seconds and the mixture was applied to root canal dentin in the post space for 20 seconds using a microbrush disposable applicator (Pentron Clinical Technologies, LLC, USA). The adhesive was dried with high pressure airflow for 5 seconds. The excess adhesive resin at the bottom of the canal was removed using a paper point and the adhesive was dried again with high pressure airflow for

further 5 seconds. The adhesives were then light-cured for 10, 20 or 30 seconds using two quartz tungsten halogen light-curing units; Optilux 501 (OP) (Demetron, Danbury, USA) and Hyperlightel (HL) (Kuraray Medical Inc, Tokyo, Japan). Their power densities, measured with a digital radiometer (Jetlite light tester, J. Morita, Mason Irvine, CA, USA), were 830 and 1350 mW/cm², respectively. The experimental groups were divided into the following six groups (three teeth for each group) according to the light-curing unit and exposure time: OP 10s, OP 20s, OP 30s, HL 10s, HL 20s, and HL 30s. All post spaces were filled with a dual-cure resin composite core material (Clearfil DC Core Automix, Kuraray Medical Inc., Tokyo, Japan). The coronal surface of the root was covered with a plastic strip and pressed gently with a glass slide to squeeze out any excess resin. Light exposure was performed for 60 seconds using the Optilux 501 by placing the tip of the light source at the top of the cavity. All specimens were then stored in water for 24 hours at 37°C.

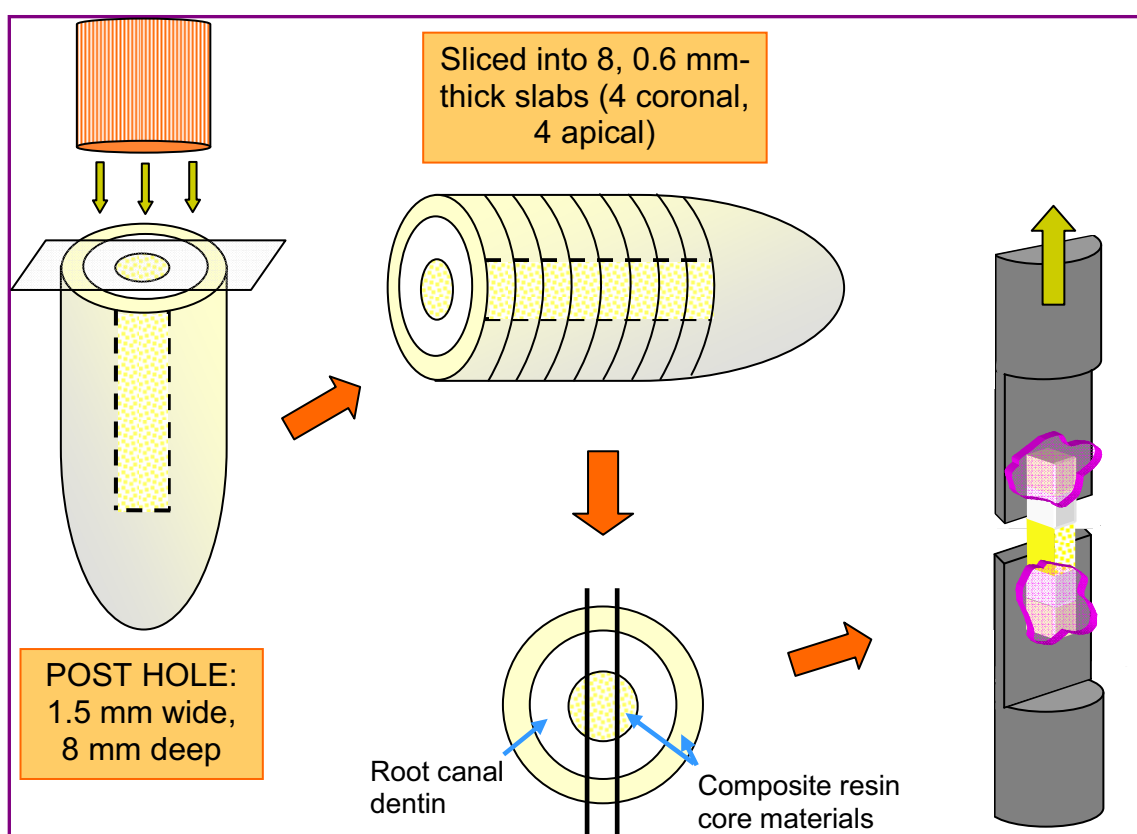


Figure 2.3 Schematic illustration of the bonding and μTBS test procedures in PART 2

Bond strength testing

After 24-hour storage, each bonded specimen was attached to the arm of a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) and eight slabs serially cut perpendicular to the bonded interface under water cooling. Each slab was then transversely sectioned at the middle part of the post cavity into approximately 0.6×0.6 mm thick beams. The cross-sectional area of each beam was measured using digital calipers (Mitutoyo CD15, Mitutoyo Co., Kawasaki, Japan). One of the two interfaces of each beam was randomly selected for testing. The ends of the beam and the remaining interface were glued onto a testing device in a table-top testing machine (EZ Test, Shimadzu Co., Kyoto, Japan) using cyanoacrylate glue (Zapit, DVA, Anaheim, CA, USA) and subjected to a tensile force at a cross-head speed of 1 mm/min.

Fracture analysis

After specimens had fractured, both the resin and dentin sides of the fractured beams were mounted on brass tablets and gold sputter-coated. Fracture modes were then observed using a scanning electron microscope (JSM—5310, JEOL, Tokyo, Japan), and classified as one of the following: adhesive failure at the dentin/adhesive interface, cohesive failure in resin including failure within adhesive or at the interface between adhesive and resin composite, mixed adhesive/cohesive failure in resin.

Statistical analysis

Microtensile bond strengths (μ TBS) of four coronal beams were considered to represent the coronal portion of the post space corresponding to the coronal third of root canal, while data of four apical beams were considered to represent the apical region corresponding to the middle third of root canal. The specimens that failed during cutting were excluded from the calculation for average bond strengths. However, the numbers of intact tested specimens were reported. The μ TBS data were analyzed using three-way ANOVA to test the effect of the light-curing unit, exposure time, and region on bond strengths. In addition, the interaction between these three factors was tested. Tukey's HSD was used as a post-hoc test for comparing 12 groups of means attributable to the presence of interactions between tested variables. The proportional frequencies of failure mode in each experimental group were compared using the chi-squared test. All statistical testing was performed at a 95% level of confidence.

PART 1 Regional bond strengths and failure analysis of fiber post bonded to root canal

Two-way ANOVA revealed that the type of fiber post had a significant effect on bond strength ($p < 0.0001$). On the other hand, regional factor had no effect on bond strength ($p = 0.100$). There was no interaction between post type and region factors in the μ TBS data ($p = 0.416$). The means and standard deviations of μ TBS for each resin post in each region are following. All values are mean (SD). The same superscripts within each row demonstrate no significant differences.

Table 3.1 Microtensile bond strength (MPa) of fiber posts to root canal dentin

	Snowlight	FibreKor	D.T. Light-Post	GC Post
Coronal	22.8(7.1) ^A	50.1(7.8) ^B	13.2(2.6) ^C	9.6(2.6) ^C
	$p > 0.05$	$p > 0.05$	$p > 0.05$	$p > 0.05$
Apical	19.9(5.5) ^a	45.3(9.5) ^b	13.4(3.6) ^c	8.4(2.3) ^c

At both regions, the highest bond strengths were obtained when FibreKor posts were used ($p < 0.05$). The μ TBS of Snowlight were significantly higher than those of D.T. Light-Post and GC Post ($p < 0.05$), whose bond strengths were similar in both the coronal and apical regions ($p > 0.05$).

The percentage of fracture modes for each post was demonstrated in figure 3.1. There were two main modes of failure: Cohesive failure within the post and failure at the post/composite resin interface. No failure at the resin composite/dentin interface was found in this study. The specimens of Snowlight all failed cohesively within the post. Approximately 75% of the GC Posts specimens also failed cohesively within the post. For FibreKor and D.T. Light-Post, failures predominantly occurred at the post/resin composite interface.

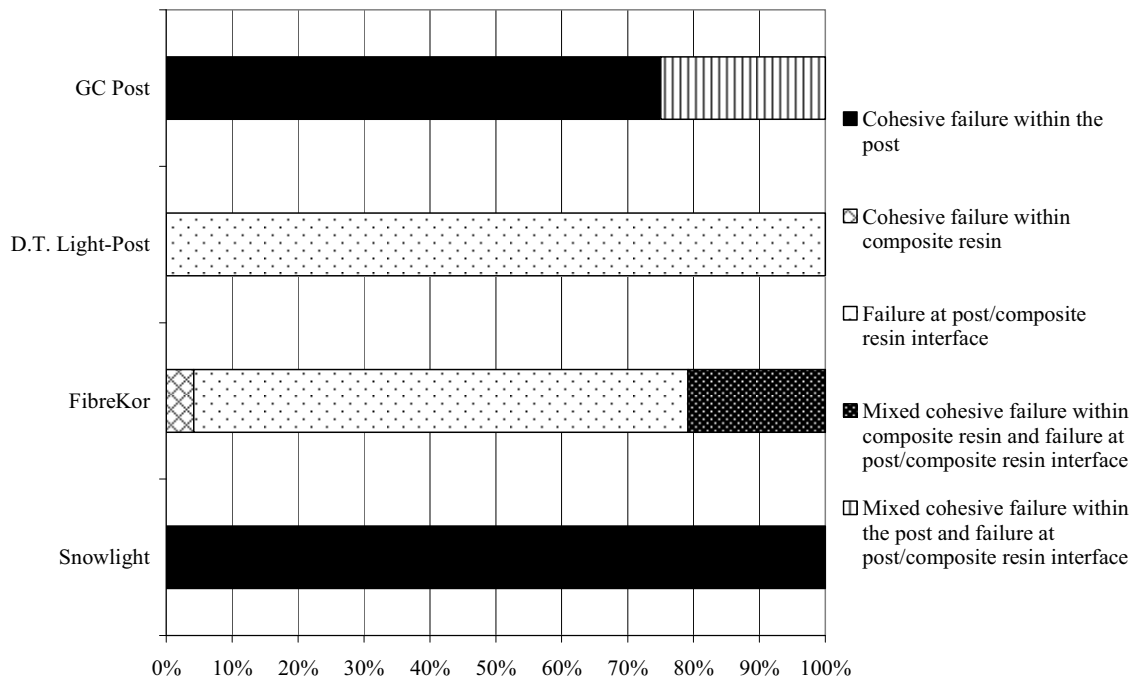


Figure 3.1 Percentage of failure mode for each fiber post system.

Representative SEM micrographs of the fractured surfaces are shown in Figure 3.2. Snowlight and GC posts mostly failed cohesively within the post, in which parallel fibers with some resin matrix in between were observed on both sides of the fractured surfaces. Mixed cohesive failure within composite resin and failure at the post/resin interface was found in FibreKor. Some fibers with fractured resin composite were observed on the post side surface and impressions of fibers (asterisks) with fractured resin were observed on the opposite resin side surface. On the post-side surface of D.T. Light-Post, stepped cut fibers were found while an impression of the post surface was observed in the resin side surface.

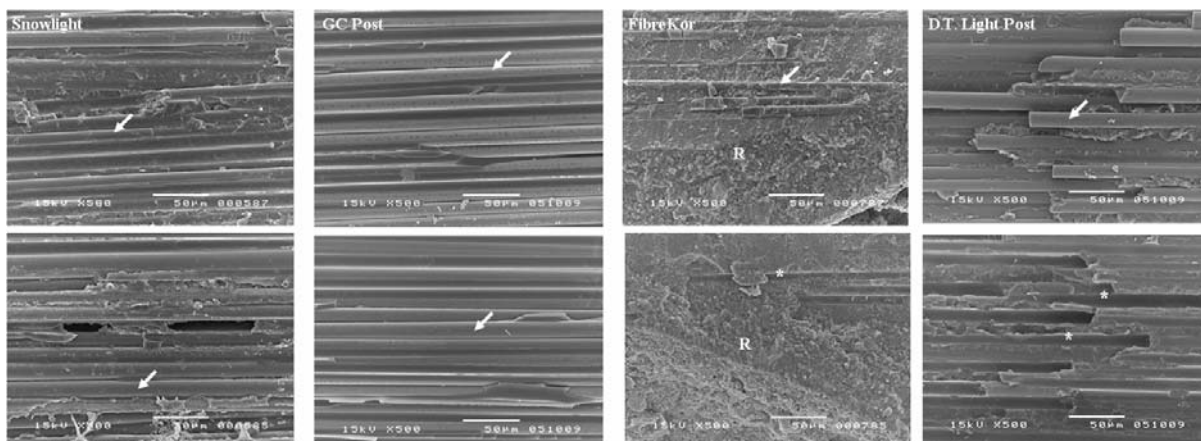


Figure 3.2 Representative SEM micrographs of the fractured surfaces.

PART 2 Effect of Photo-curing Strategy on Bonding of Dual-cure One-step Self-etch Adhesive to Root Canal Dentin

Three-way ANOVA revealed that differences in the light-curing unit ($p=0.005$), exposure time ($p<0.0001$), and region ($p<0.0001$) had a significant effect on the μ TBS. Interactions between the light-curing unit and exposure time ($p=0.011$), and between the light-curing unit and region ($p=0.048$) were also present. Regional microtensile bond strengths of dual-cure one-step self-etch adhesive to root canal dentin in each experimental group are summarized in Table 3.2. All values are mean (SD). The numbers of tested specimens are shown in the parentheses below bond strength values. NS and the same superscripts demonstrate no significant differences

Table 3.2 μ TBS (MPa) of DC Core Automix to root canal dentin treated with Clearfil DC Bond

	Optilux 501			Hyperlightel		
	10 s	20 s	30 s	10 s	20 s	30 s
Coronal	38.9(13.7) ^{AB} (11/12)	39.8(12.5) ^{AB} (12/12)	38.5(10.0) ^{AB} (12/12)	33.9(11.3) ^A (11/12)	49.8(8.6) ^B (12/12)	42.7(11.4) ^{AB} (11/12)
	$p<0.05$	$p<0.05$	NS	$p<0.05$	NS	NS
Apical	11.1(2.1) ^a (8/12)	20.4(8.3) ^{ab} (12/12)	28.6(11.1) ^{bc} (10/12)	16.0(7.2) ^{ab} (10/12)	36.4(13.3) ^c (12/12)	30.2(10.6) ^{bc} (11/12)

Although post-hoc comparison was performed in order to compare 12 groups of means, significant differences in the data as shown in Table 3.2 have been partially indicated simply to present the differences between curing methods in each region, and between regions in each curing. At the coronal region, the highest average bond strength was obtained from the HL 20s group. However, no significant differences among experimental groups were indicated ($p>0.05$) except between the HL 10s and HL 20s groups ($p=0.023$). At the apical region, high bond strengths could be obtained from the groups OP 30s, HL 20s, and HL 30s, which significant differences were not found between these three groups ($p>0.05$). The lowest bond strength occurred when adhesive resin was cured with the Optilux 501 for only 10 seconds. There were no significant differences between coronal and apical regions when the adhesive resin was cured with the Optilux 501 for 30 seconds, or the Hyperlightel for 20 and 30 seconds ($p>0.05$).

The percentage of fracture modes of the debonded specimens in each experimental group are shown in figure 3.1. There was no cohesive failure within the dentin substrate. Chi-square testing revealed no significant differences in the failure modes between each group ($p>0.05$) except the HL10s group, of which approximately 60 % of the specimens failed at the interface between the dentin and the adhesive resin.

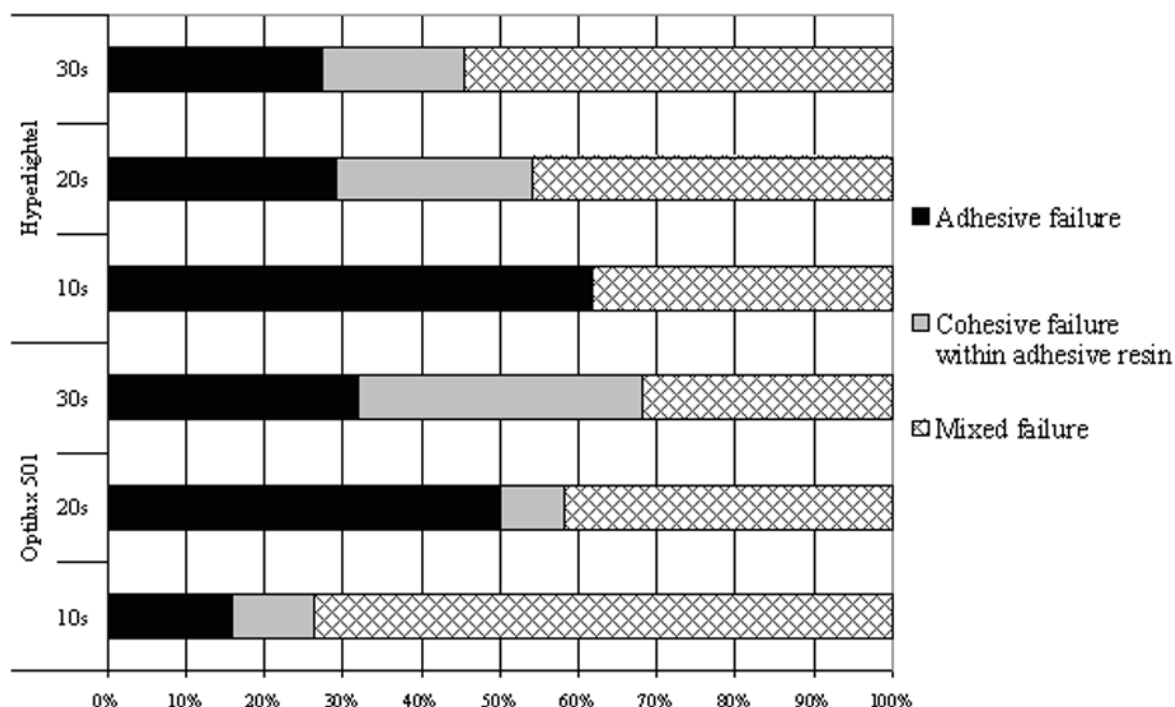
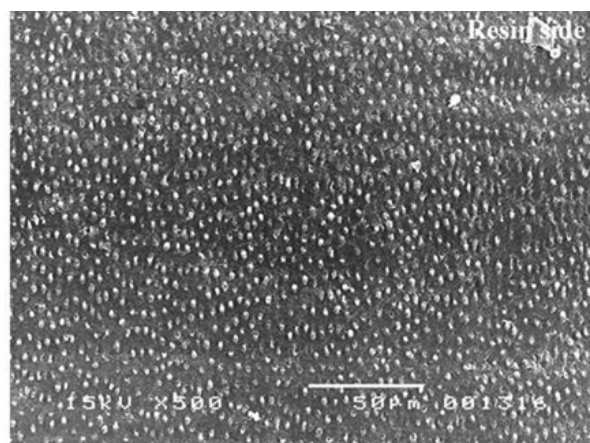
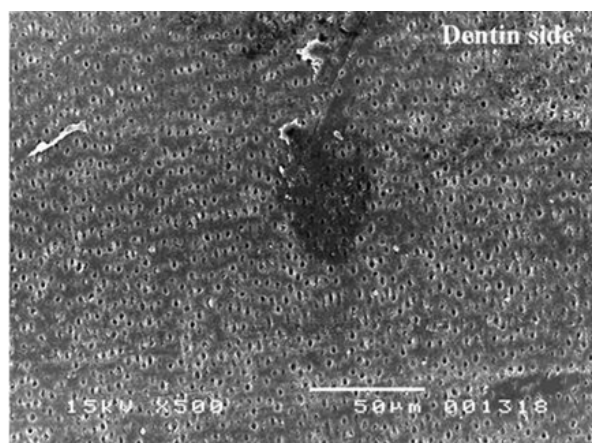
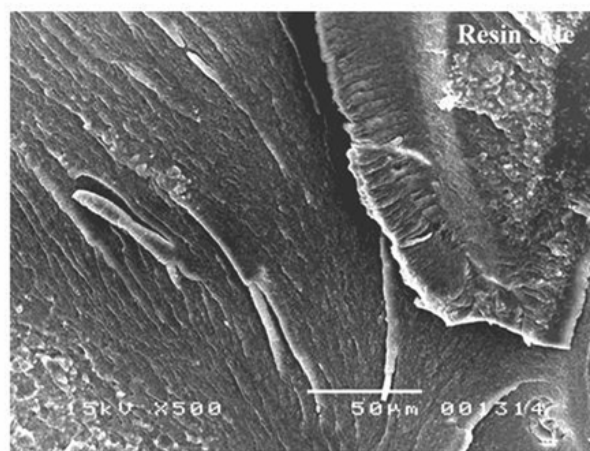
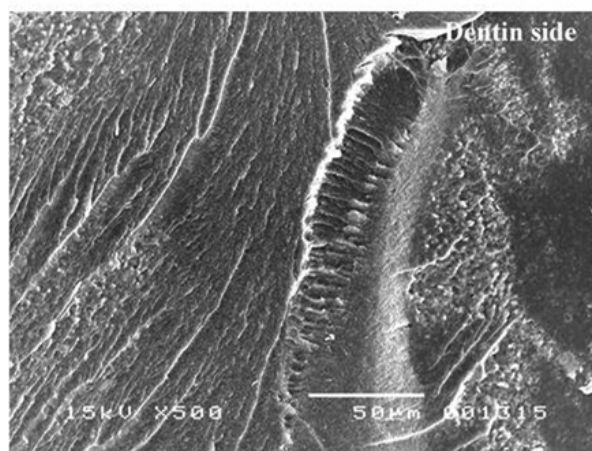


Figure 3.1 Percentage of failure mode for each curing machine and exposure time.

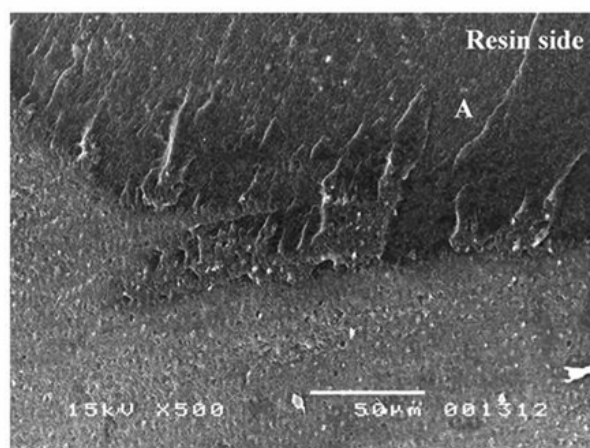
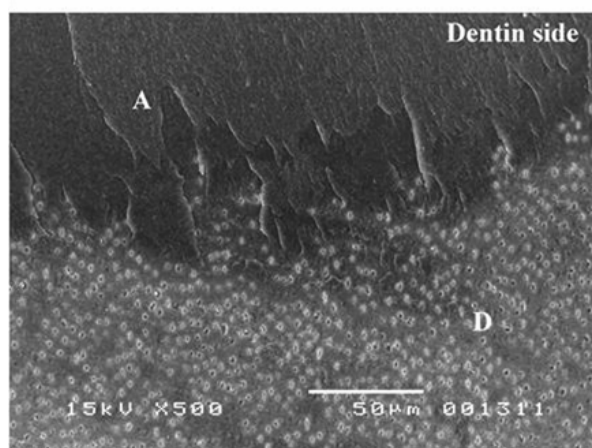
Representative SEM micrographs of the fracture surface are shown in Figure 3.2. For adhesive failure, exposed dentinal tubules were observed on the dentine side, whereas resin tags were pulled out and observed on the resin side. The cohesive failure within the resin was mostly found in the adhesive as a layering pattern. Some of the debonded specimens revealed abundant blisters at the adhesive resin surface and composite surface as shown in Figure 3.3, which depicts fracture surfaces at the apical region of the OP 10s group.



Adhesive failure



Cohesive failure in resin



Mixed adhesive failure/cohesive failure in resin

Figure 3.2 Representative SEM micrographs of the fracture surfaces of both dentin and resin sides in each failure mode