Original Article
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A comparison of the push-out bond strength between dual polymerized core build-up composite and total-etch resin luting cement for prefabricated fiber post

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Abstract

Objectives: Compare the push-out bond strength of a dual-cured core build-up resin composite and dual-cured resin cement using total-etch adhesive for bonding fiber post to root canal dentin.

Materials and Methods: Sixty extracted single-rooted human premolars were sectioned transversely in mesiodistal direction 2 mm coronal to cementoenamel junction, standard endodontically treated, post space prepared and randomly divided into two groups (n=30). Group 1 fiber posts were luted with a total-etch resin cement (Variolink® II) and group 2 were luted with a composite resin core build-up material (Luxacore® Z-Dual) using total-etch adhesive system. All roots were cut transversely into 3 sections (coronal (L1), middle (L3) and apical (L5)) with 1 mm thicknessin each section. The push-out test was performed at a speed of 0.5 mm/min. Failure modes were evaluated using a scanning electron microscope (65x). The data were analyzed using ANOVA and post hoc Tukey's test (p<0.05).

Results: In 3 root canal regions, the mean push-out bond strength of Luxacore® Z-Dual showed significantly higher bond strength than Variolink® II ($p<0.05$). Means push out bond strength of both Luxacore® Z-Dual and Variolink® II at cervical region were higher than those of middle and apical regions ($p<0.05$). The analysis of failure modes revealed that most of the failures were adhesive failure.

Conclusion: Regional push-out bond strength of Luxacore® Z-Dual resulted in significantly higher bond strength than Variolink®II and this method could be considered as an alternative technique to luted fiber post within root canal.

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Key words: *core build-up material; polymerization; push-out bond strength; resin cement; total-etch adhesives*

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Introduction

In recent years, the choice of materials used in the restoration of endodontically treated teeth has changed from rigid materials (stainless steel, gold, and zirconia) to fiber post which has modulus of elasticity similar to that of dentin (18.6 GPa). It was thought that forces would be distributed more evenly in the root, resulting in fewer root fractures (Boschian Pest et al., 2002). The previous study showed that fiber post used in combination with adhesive agent and resin cement could increase bond strength and fracture resistance when compared to that used with conventional luting cement for post bonding (Stamatacos and Simon, 2013).

Although it was found in the study that the usage of fiber post in combination with adhesive systems and resin cement would reduce the vertical root fracture, However the frequent failure pattern is the debonding of post-resin-dentin interface (Cagidiaco et al., 2008; Mosharraf and Haerian, 2011). Therefore, the efficiency of adhesive systems is an essential factor for a good bonding which affects bond strength.

From the study of Pereira et al., it was found that there were many factors which affected the long-term bond strength of an adhesive interface such as light intensity, polymerization, c-factor between adhesive and cement, the presence of bubbles and gaps in the cement layer, the anatomical and morphological characteristics of the dentin substrate, humidity control inside a root canal and the compatibility between adhesive systems and resin cements (Pereira et al., 2013). Therefore, the bonding of post inside root canal is a crucial factor in the process of ensuring the retention, marginal seal and durability of indirect restorations (Stamatacos and Simon, 2013).

The adhesive systems can be categorized into two groups according to the application steps: total-etch adhesive system and self-etch adhesive system. The total-etch adhesive system uses a 30-40 percent phosphoric acid to etch dentin and enamel, followed by the primer and adhesive resin (Van Meerbeek et al., 2005). For the self-etch adhesive system, the steps of using phosphoric acid and water rinsing are eliminated, but uses the self-etching primer that contained acidic monomer as a mixture which, however, cannot completely dissolve the smear layer (Tay and Pashley, 2001). It was found in the study that total-etch adhesive system can completely dissolve the smear layer and the inorganic compound in the dentin which results in the opening of dentinal tubules (Hayashi et al., 2008). This then allows adhesive to penetrate the opened tubules, causing hybrid layer of 5-10 micrometer thickness including the forming of resin tags of 100 micrometer (Boschian Pest et al., 2002; Hayashi et al., 2008). Such long resin tags with intertubular anastomose in their lateral branches can counter the stress caused by the polymerization shrinkage and partially contribute to enhance the mechanical bond strength (Hayashi et al., 2005). In addition, acidic resin monomers from two-step total etch and self-etching primer adhesives may impair the polymerization of dual cured cements and composites that are initiated via aperoxide-amine binary redox system. The remaining acidic monomers in oxygen-inhibition layers of adhesives may interact with tertiary amine, hence reduce the self-cured setting reaction and cause incomplete polymerization of dual-cured resin cement (Cheong et al., 2003). However, problems on technique sensitivity may be found in the total-etch adhesive; for example, it is rather difficult to perform and complete the steps of tooth conditioning with phosphoric acid and water rinsing (Van Meerbeek et al., 2005).

Resin cement comprises 2 main components: resin matrix and filler. The resin cement can be categorized into three main groups according to the bonding system: the total-etch resin cements, self-etch resin cements and self-adhesive resin cements. If it is grouped according to the polymerization mechanism, there are three groups: chemical or self polymerization, light polymerization and dual polymerization. Dual polymerization contains self-curing components to initiate a polymerization reaction in the absence of light such as within the root canal (Arrais et al., 2008). Resin cements have high bond strengths, high modulus of elasticity, high tensile and compressive strengths but disadvantages of resin cements are technique sensitivity (Stamatacos and Simon, 2013).

Although resin cements have high modulus of elasticity but they still have a lower modulus of elasticity than dentin and post thus a zone of highly concentrated loads and stressed is created especially in a flared root canal which create thick resin cement layer between post and root canal resulting in fewer bond strength and mechanical property. To overcome this problem, a dual-cured resin composite has been introduced as a core build-up material and luting agent (Boschian Pest et al., 2002; Bouillaguet et al., 2003) which has a higher modulus of elasticity than resin cement and has a modulus of elasticity close to dentin and fiber post (Pereira et al., 2013), therefore it enables resistance to occlusal stresses, and occlusal forces would be transferred from restoration to tooth structure and promotion of monoblock in root canal and in pulp chamber (Boschian Pest et al., 2002). These considerations suggest that resin composite would be a better material to use in luting fiber post, especially if the space exceeds 0.3 mm as the resin cement could lessen bond strength (D'Arcangelo et al., 2007). However, the contrastive study on bond strength of luting agents and other studies reported conflicting results (Putignano et al., 2007; Magni et al., 2007).

This study aimed at comparing between the technique of fiber post bonding with Variolink® II and Luxacore® Z-Dual. Both materials were used in combination with similar adhesives which are classified as the total-etch system. The objective of this study was to compare push-out bond strength of dual-cured core build-up resin composite and dual-cured resin cement using total-etch adhesive for bonding fiber post to root canal dentin.

Materials and Methods

This study obtained approval for the research methodology at the Human Research Ethics Committee in the Faculty of Dentistry Srinakharinwirot University (license number 31/2557). Sixty human mandibular premolar teeth were extracted not exceeding 6 months and stored in distilled water at the 4°C. The inclusion criteria were single root, fully developed apices, absence of caries or root cracks, absence of previous endodontic treatments, posts, or crowns and a root length of 16 mm from root apex to buccal cementoenamel junction. After passing all the criteria, all teeth were taken x-ray photographs for the root canal condition examination.

The crown of each tooth was removed 2 mm coronal to cementoenamel junction with a high speed diamond cylinder bur. Endodontic treatment was done using a standardized step-back technique. The apical preparation was maintained at file size 35 and obturated with lateral condensation technique using gutta-percha/AH Plus sealer (AH Plus®, Dentsply DeTrey GmbH, Konstanz, Germany). The root canal access was temporarily filled with Cavit-G (Cavit™G, 3M ESPE AG, Seefeld, Germany) for 4 mm, and teeth were stored in 100% humidity at 37°C for 24 hours.

Post space was prepared to depth of 14 mm with Peeso reamer and D.T. Light post drills no. 1 and no. 2 respectively. Irrigation was performed by distilled water. Next, D.T. Light post no. 2 (D.T. Light-Post® ILLUSION™ X-RO, R.T.D., St. Egreve, France) was tried and tooth was embedded at the depth of 4 mm from apex in self-cured acrylic resin using paralleling surveyor in order to set the right angle between long axis and horizontal plane. The prepared root canals were finally flushed with 17% EDTA, 2.5% NaOCl and distilled water respectively, and then dried with absorbent paper points.

Table 1 Composition and application modes of the materials used in the study

Luting agent	Adhesive system	Composition	Application procedures
Variolink® II	Syntac system and heliobond	**Variolink® II** - Monomer matrix: Bis-GMA, UDMA, TEGDMA - inorganic fillers: barium glass, ytterbium trifluoride, bariumaluminiumfluorosilicate glass and spheroid mixed oxide - additional contents: catalysts, stabilizers, pigments **Syntac system** - Etchant: 35% H_3PO_4 - Syntac primer: maleic acid, TEGDMA, acetone, water - Syntac adhesive: PEGDMA, glutaraldehyde, water - Heliobond: Bis-GMA, TEGDMA, stabilizers and catalysts	- Apply etchant to dentin 15 s - Rinse, remove excess water with paper points and gently dry - Apply dentin syntac primer 15 s and remove excess water with paper points and gently dry - Apply syntac adhesive 10 s and remove excess water with paper points and gently dry - Apply heliobond and remove excess water with paper points, light-cure for 20 s - Mix base and catalyst of Variolink® II in 1:1 ratio for 10 s - Carry mixed paste with lentulo spiral and seating the post into the root canal - Light-cure for 40 s
Luxacore® Z-Dual	Adper Scotchbond Multi-Purpose	**Luxacore® Z-Dual** - Bis-GMA, UDMA, TEGDMA, Barium glass, pyrogenic silicic acid, zirconium oxide **Adper Scotchbond Multi-Purpose** - Component 1 (etchant): 35% H_3PO_4 - Component 2: (Scotchbond Multi-Purpose primer) HEMA, polyalkenoic acid polymer, water - Component 3: (Scotchbond Multi-Purpose adhesive) Bis-GMA, HEMA, tertiary amines (both for light-cure and self-cure initiators), photo-initiator	-Apply etchant to dentin 15 s - Rinse, remove excess water with paper points and gently dry 2 s - Apply two layer of Scotchbond Multi-Purpose primer and remove excess water with paper points and gently dry for 5 s - Apply Scotchbond Multi-Purpose adhesive and light-cure for 10 s - Apply the automixed paste with the aid of root canal tip and seat the post into the root canal - Light-cure for 20 s (self-cure 5 min)

Information provided by manufacturers. HEMA: 2-hydroxymethyl methacrylate, Bis-GMA: bisphenylglycidyl dimethacrylate, UDMA: urethane dimethacrylate, PEGDMA: poly(ethylene glycol) dimethacrylate, TEGDMA: tri-ethylene-glycol dimethacrylate

All teeth were randomly divided into 2 groups with 30 tooth roots in each group and luting procedures were performed. In group 1, posts (D.T. Light-Post® ILLUSION™ X-RO, RTD, St. Egreve, France) were bonded with Variolink® II using syntac system and heliobond (Ivoclar Vivadent, Schaan, Liechtenstein). In group 2, posts were bonded with Luxacore® Z-Dual (DMG, Hamburg, Germany) using Adper Scotchbond™ Multi-Purpose (3M ESPE, St. Paul, MN, USA). The luting procedures performed in each experimental group, and the composition of the materials used in the study are reported in Table 1. All roots were stored in 100% humidity at 37°C for 24 hours.

A low-speed diamond saw (ISOMET 1000, Buehler, Lake Bluff, IL, USA) was used to section each root horizontally. Firstly, the cut at 4 mm located from coronal to apical was not used, but the other five pieces, L1-L5, were equally cut of 1 mm thickness. Three pieces were used as samples. L1 was used as sample of coronal part, L3 was used as sample of middle part, and L5 was used as sample of apical part (Fig. 1). Push-out test was performed by applying a compressive load with a Universal testing machine (EZ Test Series, Shimadzu, Kyoto, Japan) at the speed of 0.5 mm/min with the load applied in the apical-coronal direction until the post segment was dislodged (Fig. 2). Because of the tapered design of the post, three different sizes of punch pin: 0.9 mm for the coronal, 0.8 mm for the middle and 0.7 mm for the apical part, was used, which was centered on the post segment and had no contact with the surrounding dentin surface. The maximum value was recorded in Newtons and converted into Megapascal for strength value with the following formula (Mumcu et al., 2010):

(MPa) = F(N)/A(mm^2) where: A = post interface area

$$A = \pi(R+r)\sqrt{\{(R-r)^2+h^2\}}$$

π = 3.1416

R = the radius of coronal part of post segment (mm)

r = the radius of apical part of post segment (mm)

h = the thickness of post segment (mm)

The thickness of post segment was measured by digital verneir caliper with a resolution of 0.01 mm. The radius of post was measured using the program Image J (Image J, Wayne Rasband & The National

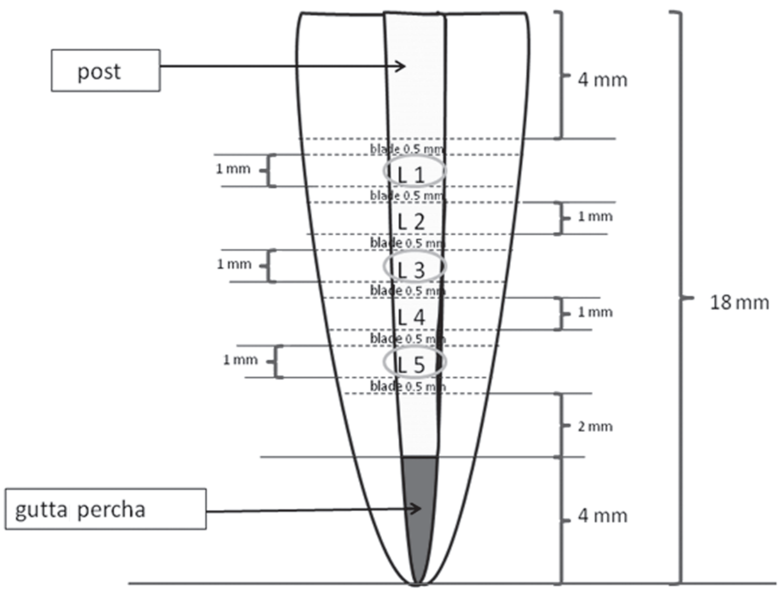

Fig. 1 Preparation of specimens for push-out bond strength testing

Institute of Health (NIH), Bethesda, Maryland, USA).

The failure mode of all specimens was evaluated under scanning electron microscope (65x). Adhesive failure was classified as the fracture either between resin cement and dentin or between resin cement and post. Cohesive failure was classified as the fracture within each substrate either post, dentin, or resin materials. In case of all failure types occurred simultaneously, mixed failure was categorized. Subsequently, the data were analyzed by ANOVA with Tukey's post hoc multiple comparison test at the significant level of 0.05

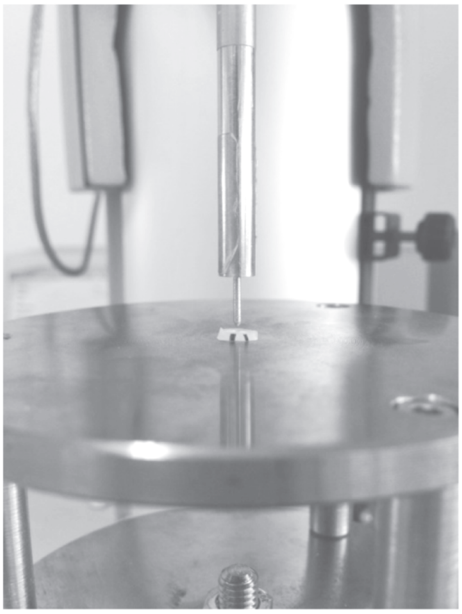

Fig. 2 The push-out bond strength test was performed using the Universal testing machine

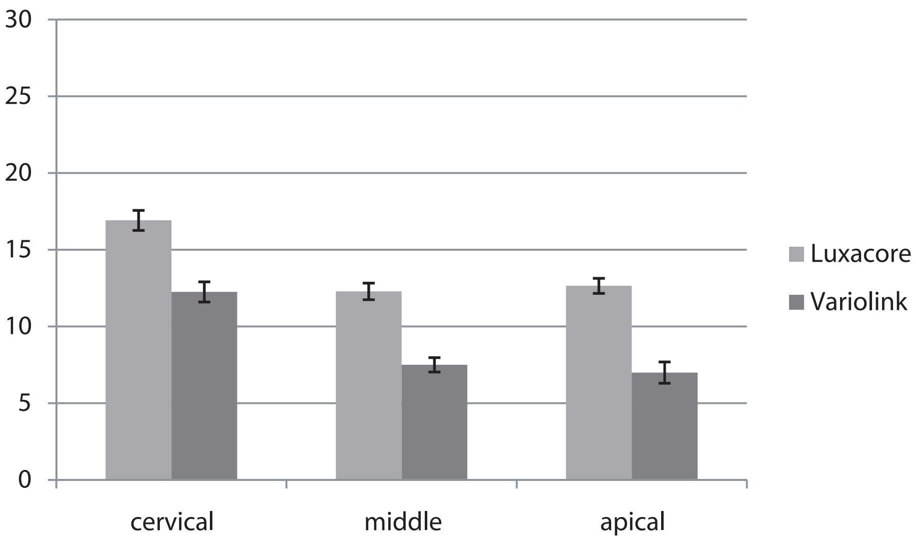

Fig. 3 Bar chart representation of mean and standard error of push-out bond strength (Megapascal : MPa)

Table 2 Mean and standard error (MPa) of push-out bond strength

Group	Mean (standard error)		
	Cervical	Middle	Apical
Luxacore® Z-Dual	16.92 (0.65)[a]	12.29 (0.54)[b]	12.65 (0.49)[b]
Variolink® II	12.26 (0.66)[b]	7.51 (0.47)[c]	7.00 (0.70)[c]

The mean difference is significant at the 0.05 level.
Different letters indicate significant difference ($p<0.05$)

Table 3 Mode of failures

Group	Root canal third	Mode of Failures (%)			
		Adhesive failure		Mix failure	Cohesive failure
		Dentin and resin cement	Resin cement and post		
Luxacore® Z-Dual	Cervical	16.67	53.33	30	0
	Middle	6.67	56.67	36.67	0
	Apical	13.33	60	26.67	0
Variolink® II	Cervical	56.67	10	33.33	0
	Middle	66.67	3.33	30	0
	Apical	50	6.67	43.33	0

Results

The mean push-out bond strengths (MPa) and standard error are presented in Table 2 and the values are shown in the bar graph (Fig. 3). From the analyzed data, it was found that, at the same level of root canal, the mean push-out bond strength of Luxacore® Z-Dual with Adper Scotchbond Multi-Purpose showed significantly higher bond strength than that of Variolink® II ($p<0.05$). For the sections level factors, the means push-out bond strengths at cervical region were higher than that at middle and apical regions ($p<0.05$), but mean push-out bond strength at middle and apical regions exhibited no statistically significant difference ($p>0.05$).

The analysis of failure modes are presented in Table 3, and samples of failure mode are shown in Fig. 4. Adhesive failure and mixed failure were found in both groups; however, cohesive failure was not found in all specimens. The most of Variolink® II specimens had an adhesive failure between root canal wall and resin cement. In Luxacore® Z-Dual group, most specimens had an adhesive failure between cement and post.

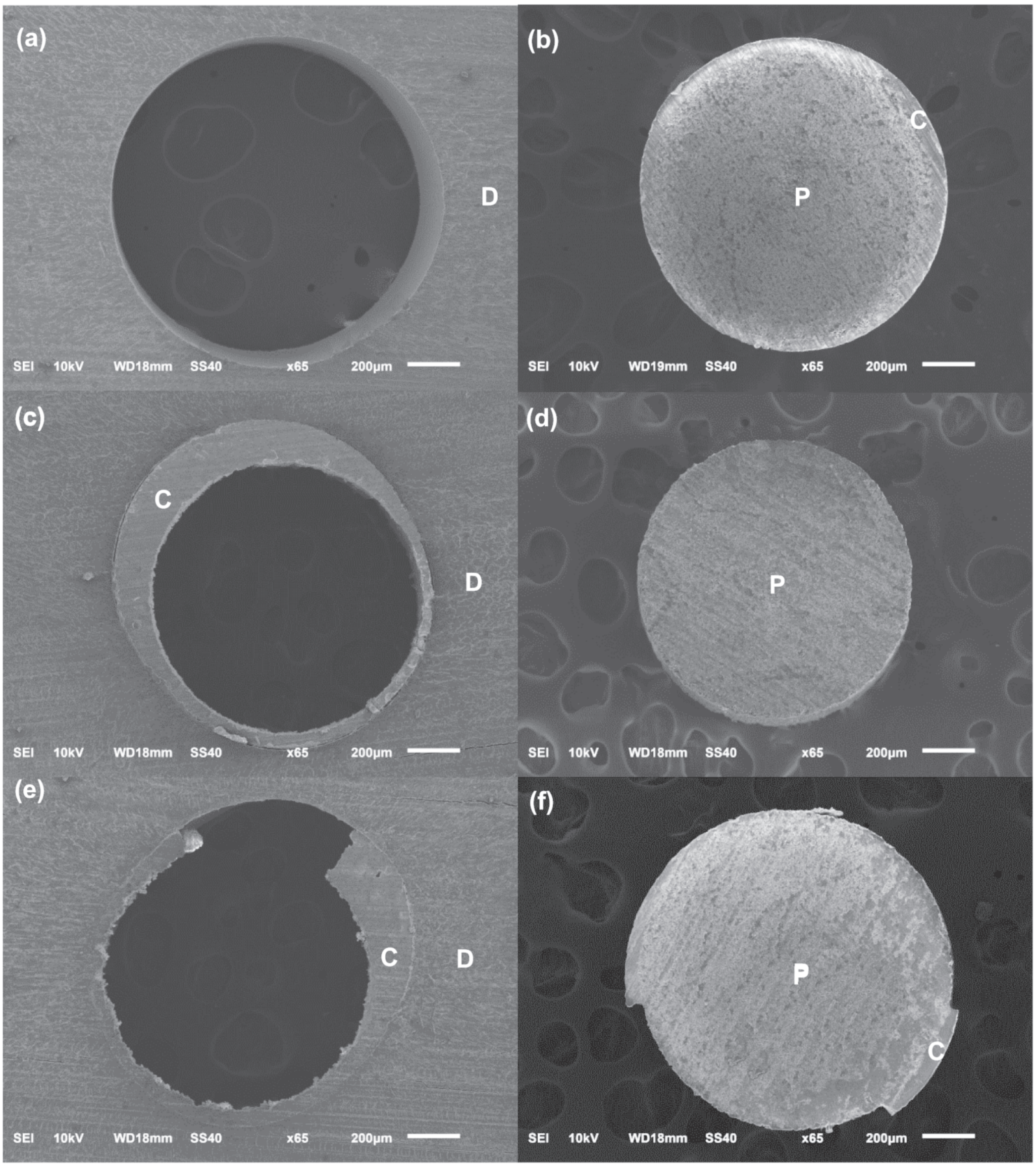

Fig. 4 SEM image of a failed specimen. (a and b); The specimen with adhesive failure between root dentin and luting agent. (c and d); The specimen with adhesive failure between post and cement. (e and f); The specimen with mixed failure (D: Dentin; C: Cement; P: Post) (Magnification x 65)

Discussion

The present study investigated the bond strengths of resin cements to root canal dentin using a push-out model. Push-out tests result in a shear stress at the interface between dentin and cement as well as between post and cement (Van Meerbeek et al., 2003), which is comparable to the stresses under clinical conditions. Although there is a speculation about the effect of stress that it seemed to contribute to the bond strengths of fiber posts using a push-out model (Goracci et al., 2005). Nevertheless, a study of Ahmadian et al. found that 1 mm thickness of push-out model provides a uniformly stress distribution (Ahmadian et al., 2013). Goracci et al. reported that the push-out test seems to be the most accurate and reliable technique to measure the bonds of fiber posts to root canal dentin compared with conventional and microtensile tests because of the absence of premature failures and the variability of data distribution. (Goracci et al., 2004).

From the result of this study, Luxacore® Z-Dual showed significantly higher bond strength than Variolink® II ($p < 0.05$) in every level of root canal which corresponded to the previous studies (Rödig et al., 2010; Raghad and Askary, 2014). This might be caused by the differences in resin matrix composition. According to the manufacturers' information, Luxacore® Z-Dual is mainly composed of UDMA (Urethane dimethacrylate), but Variolink® II is mainly composed of Bis-GMA (Bisphenylglycidyl dimethacrylate). From the previous studies, it was found that the structure of UDMA had weak hydrogen bond causing high flexibility and molecule movement in the polymer chain. This resulted in increasing rate of polymerization which affected the high bond strength, so encouraged a better mechanical and physical property of UDMA than the Bis-GMA (Barszczewska-Rybarek, 2009; Gajewski et al., 2012). Besides, Luxacore® Z-Dual is used in the form of automix causing static mixture and fewer void in resin when compared to the manual mixing in Variolink® II (Aksornmuang et al., 2007; Raghad and Askary, 2014). These voids would be incorporated into the material and lead to numerous oxygen-inhibition zones of unpolymerized materials, which then affect the strength and solubility of the resin cement. The latter then leads to debonding and/or fracture of the restoration (Aksornmuang et al., 2007; Mese et al., 2008). Moreover, some type of adhesive system may affect the bond strength such as Variolink® II used with classic syntac system. Conditioning dentin step of classic syntac system composes of phosphoric acid etch (remove smear layer, expose collagen and tubules) and syntac primer (modify smear layer, expose both collagen and dentinal tubules). The characteristic of this double etching cause over-etching which have dissolved residual hydroxyapatite in the heavily demineralized peri-and intertubular dentin, resulting in lower bond strength (Schmidlin et al., 2008).

According to the differences of solvent used in adhesive system. it was found that classic syntac system comprised an acetone-based adhesive which bonded effectively with wet dentin (Manso AP et al., 2008). A study of Hashimoto M et al. found that the infiltration ratio of the bonding resin within the hybrid layer for acetone-based systems was reduced by 50% when applied to over-dried dentin (Hashimoto M et al., 2002). The root canal may happen slightly overdrying condition. From this reason, Variolink® II used with classic syntac system showed lower bond strength. While Luxacore® Z-Dual used with Adper Scotchbond™ Multi-Purpose which comprised an alcohol-based adhesive has been reported that it worked better in over-dried dentin (Manso AP et al., 2008). So, Luxacore® Z-Dual showed higher bond strength than Variolink® II. However, previous studies found that there were no statistically significant differences in push-out bond strengths between root canal restored with resin composite as core build-up material and resin cement (Putignano et al., 2007; Magni et al., 2007).

From this study, the push-out bond strengths in coronal region had significantly higher than middle region and apical region both in Variolink® II group and Luxacore® Z-Dual group which corresponded to the previous studies (Wang et al., 2008; D' Arcangelo et al., 2008). Because, in the coronal region, there was greater number and diameter of dentinal tubule than middle and apical regions (Vichi et al., 2002; Topcu et al., 2010) causing more penetration of the adhesive, more hybrid layers and also more density of the resin tag. (Ferrari M et al., 2000). Besides, total-etch adhesive had efficiency of removing smear layers and dissolving inorganic components in root canal dentin resulting in good micromechanical retention of the resin cement and dentin (Boschian Pest et al., 2002). A study of Sirimongkolwattana et al. found that total-etch resin cement in the coronal region caused long resin tag and dense hybrid layer more than the middle and apical region causing higher bond strength in the coronal region (Sirimongkolwattana et al., 2014). Moreover, the coronal region was better accessibility, so it is easy to surface conditioning the root canal dentin with acid and adhesive agents. (Bouillaguet et al., 2003; Perdigao et al., 2007) In addition, as the coronal region was located near the light source, better light polymerization could occur, which corresponded to the study of Aksornmuang et al. who found that resin composite in the coronal region polymerized through photo-activation resulting in higher ultimate tensile strength, whereas resin composite in the apical region polymerized mainly through chemical activation. Thus, the polymerization of resin composite in coronal part was higher than that in the apical area (Aksornmuang et al., 2007).

The resin cement used in this study is dual-cured and the fiber post used in this study has a light transmission property. However, Teixeira et al., found that only 22 percentages of light intensity could penetrate through D.T. light post at the distance of 10 mm (Teixeira et al., 2006). Besides, the study of Goracci et al. reported that light intensity was significantly decreased from coronal to apical region causing incomplete polymerization in the apical area which affected the lower bond strength (Goracci et al., 2008).

The push-out bond strengths in the apical area should have been less than that in the middle area due to the factors in the reduction of light intensity, the decreasing in number and diameter of dentinal tubules, the difficulty in the application of the adhesion protocol affecting the flowing ability of resin cement (Topcu et al., 2010; Mumcu et al., 2010). At the apical area there was the remmants of sealer and gutta-percha which hinder the bonding procedure (Muniz and Mathias, 2005). Moreover, there are other conditions that are less favorable for bonding and causing non-uniform hybridized dentine (Vichi et al., 2002), such as irregular secondary dentin, cementum-like tissue on the root canal wall and accessory canals (Mjör et al., 2001). In addition, the apical structure has the poor collagen condition which is generally changed by bacteria and its enzyme causing alteration of dentin property (Mjör et al., 2001). Surprisingly, the results revealed that the push-out bond strength in the apical area was not different from that in the middle area. From the compensation of various factors, it can be explained that there was reduction of light intensity in the apical area causing less light polymerization and also reduction of contraction stress. As a result, the resin composite has more time for stress relief and polymerizes with self-cured mode which affected the increasing bond strength (Braga et al., 2002; Bouillaguet et al., 2003). Moreover, at the apical area, there was a thin layer of resin cement or core build up material between post and root canal wall which the thin layer of resin cement was produced from precisely post space preparation by using preformed D.T. drill, therefore it closely adapted to post surface and root canal wall. This increased bond strength which corresponded to the study which found that the bonding between fiber post and root canal depended more on the frictional sliding resistance against the dislodging of post than micromechanical and chemical adhesion from luting material (Goracci et al., 2005). However, there were a conflicting studies finding that there was no significant difference in the bond strength in each area of root canal (Foxton et al., 2005; Aksornmuang et al., 2008). Furthermore, a number of studies have reported there was higher bond strength in the apical region than in middle and coronal regions (Muniz and Mathias, 2005; Bitter et al., 2006).

Regarding to the mode of failure, it was found in this study that Variolink® II had adhesive failure between dentin and luting material more than other types which corresponded to the previous studies (Teixeira et al., 2006; Kadam et al., 2013). The adhesive failure between dentin and luting material was resulted from the factors which affected the bond strength of an adhesive interface such as conditioning dentin step by using phosphoric acid and by using syntac primer in classic syntac system. A repeated dentin conditioning step from phosphoric acid and syntac primer introduces defects at the interface between dentin and bonding. The study of Belli R et al., showed that the fracture patterns of syntac occurred mainly of adhesive nature, between the adhesive resin and dentin interface (Belli R et al., 2010). Meanwhile, in the Luxacore® Z-Dual group adhesive failure was found between post and luting agent more than others which corresponded to the previous studies (de Durâo Mauricio et al., 2007; Raghad and Askary, 2014). This reason might be due to no pretreatment of the post surface causing failure between post and luting agent.

In this study, the experiment was carried out on human teeth each of which normally contained variety of structure components even in different areas of the same tooth. The existence of different characteristics of tooth is an uncontrollable factor, and these factors might affect the value of push-out bond strength.

One limitation of this study is that it was an *in vitro* study which could not fully simulate oral conditions. In addition, this study presented outcomes limited to a single-loaded test; thus, further studies should include thermal, load cycling, and aging methods to challenge an adhesive interface. Finally, all proposed luting cements and all adhesive strategies for bonding fiber post to root dentin merit further clinical trials to investigate their long-term clinical performance.

Conclusions

Within the limitations of this *in vitro* study, it can be concluded that the type of luting agent affected the bond strength. It was found that the value of push-out bond strength in bonding fiber post with total-etch adhesive in Luxacore® Z-Dual was significantly higher than Variolink® II at every level of root canal ($p < 0.05$). Moreover, push-out bond strengths both Luxacore® Z-Dual and Variolink® II groups at cervical region were higher than at middle and apical regions ($p < 0.05$). However, there was no statistically significant difference of push-out bond strength at middle and apical regions ($p > 0.05$).

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