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Original Article

Effects of resin cements on hardness and thickness around titanium post: an intraradicular assessment

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Abstract The purpose of this study was to evaluate hardness (indicator for polymerization) and thickness of two types of resin cement at coronal, middle and apical level of tooth root canal. Ten extracted maxillary incisors were instrumented and post space was prepared for cementation of titanium post. Samples were divided into two groups and each group was cemented either of the two types of resin cements; Panavia F [dual-cured (PF)] and Rely X Luting 2 [self-cured (RL)]. The teeth were longitudinally sectioned; hardness and thickness was measured using Vickers hardness tester and a microscope (Leica DMLM). SEM observations along the cement line at the 3 different root levels were performed. Statistical analysis was performed to test significance of differences in hardness and thickness of the two types of cement (*t*-test; $p= 0.05$) and at different levels of the same type (one-way ANOVA followed by multiple comparison; $p= 0.05$). Significant difference of hardness was found at the apical level between the two groups and between the coronal and apical level of PF ($p<0.05$); no significant differences in hardness and thickness were observed in RL ($p>0.05$). Moreover, voids were more obvious within the dual-cured group of cement. Dual-cured resin cement was found to be less polymerized than self-cured type at apical level. Increased thicknesses of resin cements in comparison to post space size were observed in both groups. Use of metallic post with resin cements needs further evaluation.

Keywords: polymerization, resin cements, titanium post.

Introduction

Endodontic posts are widely used to restore teeth with insufficient coronal tooth structure to retain a core for a definitive restoration (Assif and Gorfil, 1994). Conventionally, zinc-phosphate cement had been used for post cementation. However, newer dental cements and bonding agents are being used in recent days, and these cements have been shown to rival or surpass the properties of zinc-phosphate cement (Tjan *et al.,* 1987). Posts bonded with resin cements had demonstrated to reinforce the restored roots and showed less microleakage than conventionally cemented posts (Kremeier *et al.,* 2008). According to the activation mode, resin cements are usually divided in three groups; photo-activated (light-cured), chemically-activated (self-cured), and dual-cured cements. Light-cured resin cements allow sufficient time and control for proper seating of the post into the canal (Yoldas and Alaçam, 2005). However, even when translucent fiber posts are supposed to transmit the light into the post space a reduction of resin cement hardness with depth has been determined (Ferrari *et al*., 2001; Roberts *et al*., 2004). Therefore, light-cured cements have indication restriction because of the severe reduction of light intensity during its transmission through the restoration (Söderholm and Reetz, 1996).

The use of self-cured resin cements guarantees polymerization without the influence of post space depth, but offers worse handling characteristics due to the absence of control of the setting reaction. Dual-cured resin cements had been developed with the objective of conciliating the favorable characteristics of self and light activated cements. These are effective control of working time and the possibility of achieving adequate degree of conversion, even in the absence of light (Braga *et al*., 2002). These cements are ideal for situations in which the opacity of the restoration or cavity depth might make it difficult for light to reach the full thickness of the cement layer (Braga *et al*., 2002). There are concerns that if light exposure is not sufficient, the light activate route of curing will be affected and maximum curing can be compromised (Rueggeberg and Caughman, 1993).

Prefabricated metallic posts made of stainless steel, nickel chromium alloy, or titanium alloys are being used. Among those, titanium has become very popular for direct post application technique. Titanium has been increasingly used in dental applications because of its excellent biocompatibility, high corrosion resistance, low density, high strength/weight ratio, high ductility, low thermal conductivity and adequate mechanical properties (Ohkubo *et al*., 2000; Zinelis, 2000). For metallic restoration, the polymerization of cements is exclusively dependent on chemical activation, since the metal forms a physical barrier to transmission of light, making photo-activation impossible. However, studies indicate that chemical activation alone is insufficient for dual-curing resin cements to achieve maximum conversion (Hasegawa *et al*., 1991; Darr and Jacobsen, 1995; El-Badrawy and El-Mowafy, 1995; El-Mowafy *et al*., 1999; Caughman *et al*., 2001).

To develop mechanical properties adequate for clinical function, resin cements must achieve proper polymerization. (Faria e Silva *et al.*, 2007). The process of polymerization occurs by conversion of the carbon double bonds that can be measured through conversion degree (Bandéca *et al*., 2009). Evidence has been found that limited degree of conversion may affect hardness and fracture toughness. In addition, inadequate cure of resin cement can also be a causative factor in postoperative sensitivity and biocompatibility problems (McComb, 1996). Besides, investigators were concerned that monomers from incompletely polymerized resin cement and adhesive may leak through the apical root filling and damage the periodontal tissue as a result of inflammatory, cytotoxic and mutagenic reactions (Faria e Silva *et al.*, 2007).

The assessment of some mechanical properties can provide an indirect measurement of the quality of polymerization of composite resins. Microhardness tests have been shown to present good correlation with Fourier infrared spectroscopy analysis (Ferracane, 1985; Rueggeberg and Craig, 1988). The surface hardness of cured resin materials can be a useful indicator of the degree of monomer conversion. Although application of titanium post have been a frequent choice for the clinicians and have become very popular, the polymerization of cementing materials had not been scientifically evaluated for metallic post. In this study Vickers hardness measurements were used as an indicator of the degree of polymerization of resin cement materials. Several authors have reported that the depth of cure of a photo-activated resin is adequate if its bottom-to-top hardness ratio is at least 0.8 (Pilo and Cardash, 1985).

Resin cements are usually applied in thin layers. A homogenous and adequate thickness of resin cement is prerequisite for good retention. Variations in the cement film thickness along the post may cause non-homogenous stress transmission through the root that might affect the failure rate of the post in the long-term (Verzijden *et al*., 1992). In addition, if the post does not fit well, especially at the coronal level, the cement layer end up being too thick, and bubbles are likely to present within it, all of which predisposes to endodontic failure. Thus, the thicknesses of dual-cured and self-cured resin cements at different root canal levels with titanium post needs to be evaluated.

The purpose of the present study was to determine hardness and thickness of dual-cured and self-cured resin cements at different depth of root canal levels using titanium post.

Materials and methods

Freshly extracted maxillary incisors with similar root length were selected for this study. The Universiti Sains Malaysia's Human Research Ethics Committee had approved this study and the teeth had been collected from dental clinics of Universiti Sains Malaysia following proper methods of the approval. External debris was removed from the teeth using an ultrasonic scaler (Electro Medical Systems S.A., Le Sentier, Switzerland). Teeth with roots shorter than 10 mm or with defects or cracks were excluded. The selected specimens were stored in normal saline solution until the start of root canal obturation. Each tooth was sectioned with a tungsten carbide rotary cutting instrument (Multi-purpose™ Bur, Dentsply Maillefer, USA) under water spray, 1 mm coronal to the cement-enamel junction. The coronal surface of each root specimen was flattened with a finishing diamond rotary cutting instrument under water spray to obtain a surface perpendicular to the long axis of the root. Root canal preparation was performed at a working length of -1mm from the apical foramen using K-files (MANI, INC, Tochigi, Japan) with step back technique. Sodium hypochlorite solution (5.25%) was used to irrigate the canals throughout instrumentation. The roots were dried with paper points and obturated with gutta-percha points by means of cold lateral condensation with eugenol free polymeric calcium hydroxide as root canal sealer (Sealapex™, Kerr Corporation, USA). Subsequently, the pulp chambers were filled with temporary filling material (Ceivitron, RECO-DENT International Co., Taiwan) and stored in normal saline (0.9%) for 24 hours. The gutta-percha was removed using Gates Glidden bur (MANI, INC, Tochigi, Japan). For cementation of titanium post, the root canals were enlarged with a low speed drill of the endodontic post system (PARAPOST® XH™, Coltene/ Whaledent Inc, USA). The depth of the post space preparation was 10 mm. Post space preparation and selection of post diameter size was done in such a way so that the difference between the size of post drill and titanium post diameter is consistent in all samples, which is 0.50 mm in diameter. Irrigation after preparation was performed with sodium hypochlorite (5.25%), and then the post spaces were dried with paper points. The samples were randomly divided into two groups of five specimens each.

Group A

Group A specimens were cemented with dual-cure resin cements (Panavia F (PF), Kurary Medical Inc, Japan) following manufacturer's instructions. Each canal was conditioned with an adhesive primer (ED primer, Panavia F, Kuraray Medical Inc.) which consist of liquid A and B. One drop of liquid A and B were mixed and applied to the post space walls with a microbrush provided by the manufacturer. Excess primer was removed with a paper point; the remaining material was gently air-dried. Equal amounts of Panavia F paste base and catalyst were mixed and applied to the post space walls and titanium post was cemented and light cured for 20 seconds. The specimens were kept in light proof environment until the moment of the test.

Group B

Specimens of group B were cemented with self cure resin cements (Rely X™ Luting 2 (RL), 3M ESPE, USA) following manufacturer's instructions. The paste was dispensed in equal volumes by fully depressing the clicker lever and mixing for 20 seconds using plastic spatula until a uniform color is achieved. A thin layer of cement was applied into the post space by using the post. The post was seated with light pressure for 2 minutes, and then excess cement was removed. After the material has completely set (after 5 minutes), it was kept in normal saline until the moment of the test.

Sectioning and Vickers hardness number (VHN) measurements

After storage in normal saline, the roots were fixed into resin block (Leocryl methyl-methacrylate, Italy) and were attached on slides. Roots were longitudinally sectioned with a diamond band saw under water irrigation (EXAKT 300 and EXAKT 310 diamond band saws, Germany). A first section was carefully made 0.30 mm from the post, so that the outermost surface of the cement layer could be exposed. Gradual polishing was done with grinding machine (EXAKT 400 CS, Germany) then with 1200-2500-grit SiC paper (Pedreira *et al*., 2009). The method of sample preparation is illustrated in Figure 1. Subsequently the samples were tested for Vickers hardness number (VHN) using Vickers hardness tester (Model VM-50, Fuel Instruments & Engineers Pvt. Ltd., Maharashtra, India). Indentations were placed at 1-mm, 4.5-mm and 8-mm from cement-enamel junction along the cement line under a static load of 1 kg for 10 seconds.

Grinding and thickness measurements

The sections were further ground using grinding machine; then with 300-1200-grit SiC paper until the post exposed. The samples were then evaluated for thickness measurements using microscope (Leica DMLM, Leica Microsystems, Bensheim, Germany). The cement thickness was measured in micrometer (μm). The quality of the resin cements of representative sample from each group were then observed using Scanning electron microscope (Phenom™, Eindhoven, Netherlands).

As the Vickers hardness and thickness data were normally distributed (Kolmogorov-Smirnov test) parametric statistical tests were selected. To test significance of differences in hardness and thickness of the two types of cements at the same level, *t*-test was performed and one-way ANOVA followed by Tukey Honestly Significant Difference (HSD) test was performed at different levels of the same group using statistical software PASW version 18 (SPSS Inc., Chicago, USA) at $\alpha = 0.05$ level.

Results

There was significant difference of cements hardness between PF and RL groups at apical level (Table 1). One way ANOVA (Table 2) showed that VHN of PF resin cements (group A) was significantly higher at coronal level compared to apical level ($p<0.05$). The VHN of apical level of PF resin cements did not achieve the 80% threshold value (bottom/top ratio) which suggests inadequate polymerization at apical region (Figure 2). For RL resin cements (group B) similar VHN at all three root canal levels was observed (Figure 3).

Thickness (μm) of the examined cements at the three root canal levels of both groups are shown in (Figure 4). Although tendency of decreased resin cements thickness was observed towards apically, one-way ANOVA revealed no significant difference of cement thickness at all three root canal levels for each group. Statistically no significant differences were found between the same root canal depths of the two groups tested ($p<0.05$). In addition, the presence of voids was observed along the cement thickness in PF comparing with RL group (Figure 5 and 6).

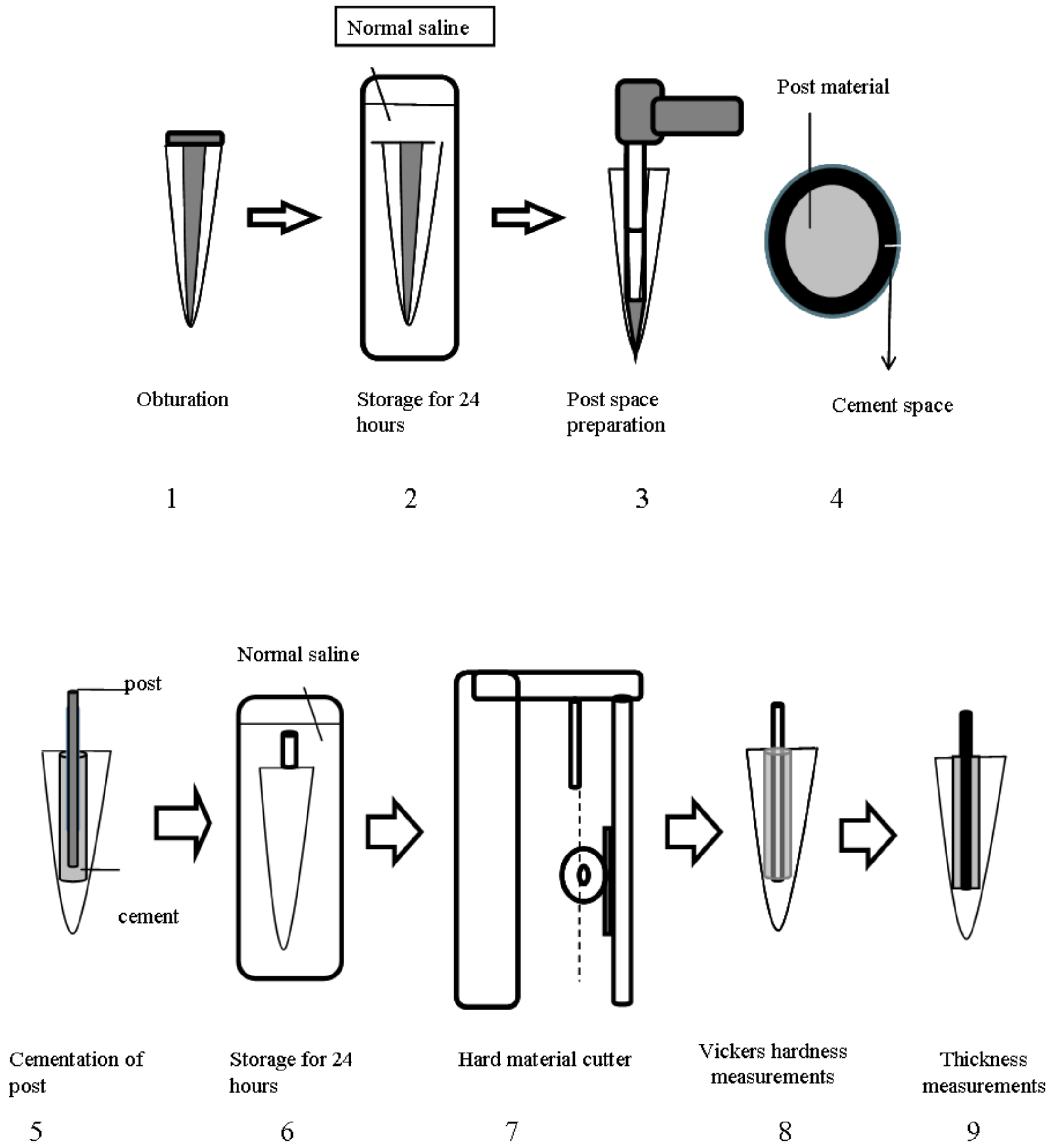

Figure 1 Schematic diagram of the sample preparation. (1) Obturation of root canal by lateral condensation. (2) Storage of specimen in normal saline for 24 hours. (3) Preparation of post space being consistent with all teeth, which is 0.5mm in diameter. (4) Cross section of a root showing the cement thickness around post material. (5) Cementation of post with resin cements. (6) Storage of specimens in normal saline for 24 hours, with group A specimens stored in light proof environment. (7) Using hard material cutter, specimen being cut at 0.30 mm from the post longitudinally. (8) Specimen polishing for hardness measurements (with no post exposed); test for hardness using Vickers hardness tester. (9) Further polishing until all post exposed; thickness measurements using Image Analyzer.

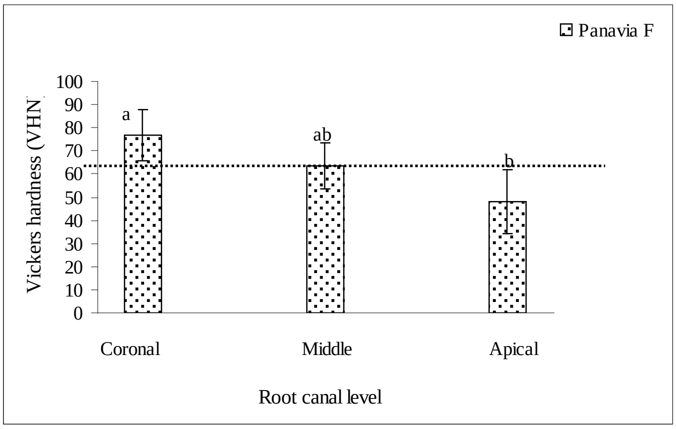

Figure 2 VHN of Panavia F (dual-cured resin cements) at three root canal levels;horizontal dotted lines represent 80% bottom/top mean Vickers Hardness ratios.

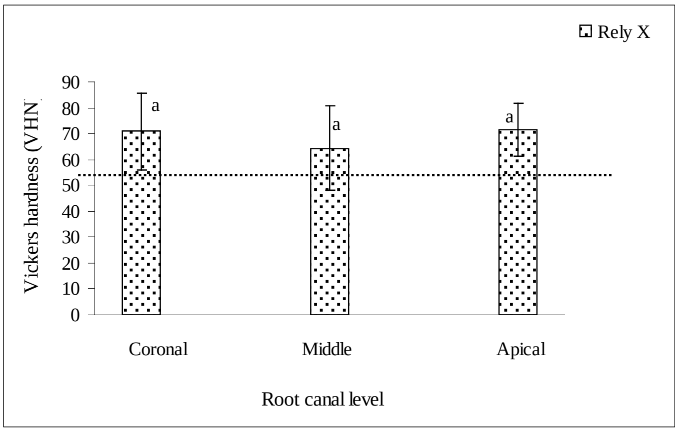

Figure 3 VHN of Rely X (self-cured resin cements) at three root canal levels; horizontal dotted lines represent 80% bottom/top mean Vickers Hardness ratios.

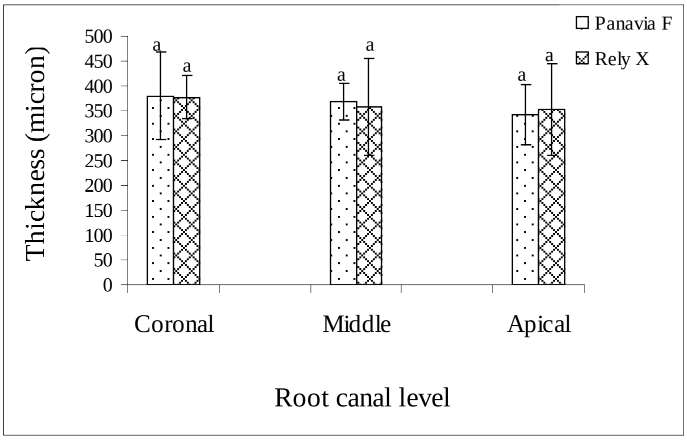

Figure 4 Thickness of dual-cured (PF) and self-cured (RL) resin cements at three different levels; independent-t test (same superscript indicates no significant difference).

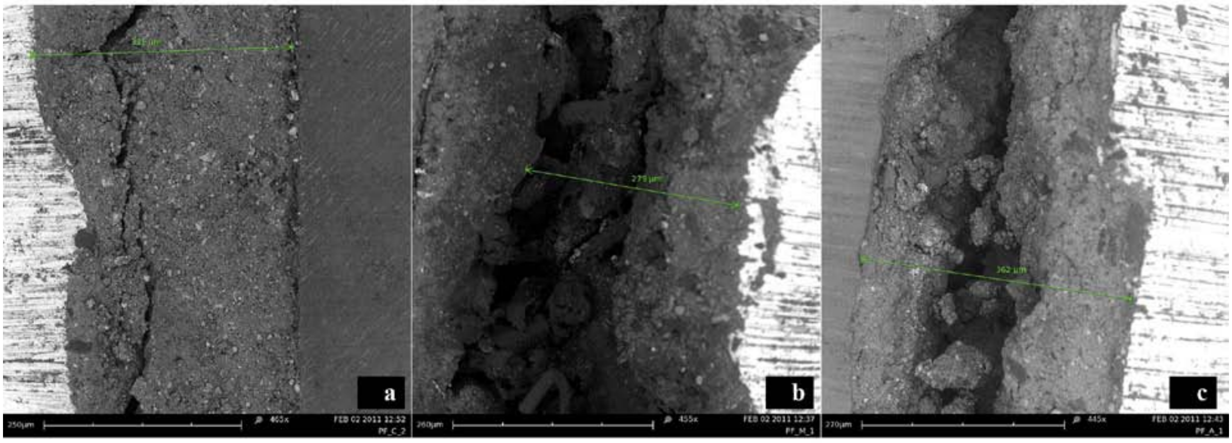

Figure 5 SEM picture of thickness of Panavia F at 3 different levels: (a) coronal, (b) middle, and (c) apical.

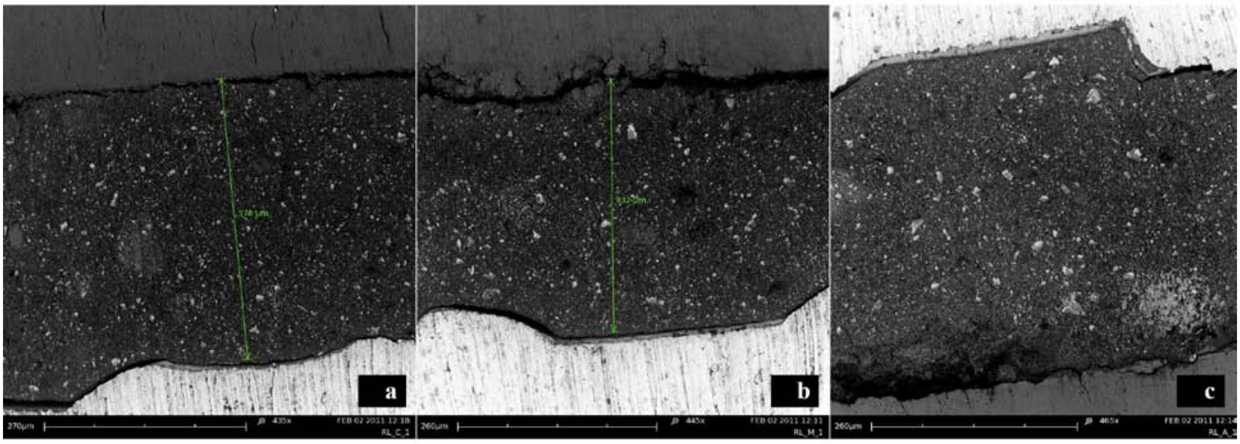

Figure 6 SEM picture of thickness of Rely X at 3 different levels: (a) coronal, (b) middle, and (c) apical.

Table 1 Comparison between PF and RL cement group between each level of root canal depth using independent *t* test

Group	Mean difference	95% confidence interval		*p value* *
		Lower bound	Upper bound	
PF coronal vs. RL coronal	6.10	-13.17221	25.37221	0.482
PF middle vs. RL middle	-.94	-21.55548	19.67548	0.916
PF apical vs. RL apical	-23.40	-41.63411	-5.16589	0.019

Independent *t*-test
* $p < 0.05$ is considered significant.

Table 2 Vickers hardness number (VHN) between 3 root canal depths of PF and RL group

		Sum of Squares	**df**	**Mean Square**	**F**	***p* value ***
PF	Between Groups	2087.957	2	1043.979	7.516	0.008
	Within Groups	1666.700	12	138.892		
	Total	3754.657	14			
RL	Between Groups	154.641	2	77.321	0.389	0.686
	Within Groups	2387.772	12	198.981		
	Total	2542.413	14			

One Way ANOVA
* $p < 0.05$ is considered significant.

Discussion

Evaluation of polymerization of resin cement around endodontic post is critical due to technique sensitivity of the available measurement methods. Thus surface hardness measurement of resin cements/ composite materials had been found to be an effective tool although strict cutting and polishing methods had to be followed for sample preparation in the present study (Barghi and McAlister, 2003; Soares *et al*., 2006; Koch *et al*., 2007). Thickness of resin cements often varies although cutting tools with recommended sizes are followed during post space preparation. The result of the present study suggests that light exposure and light transmitting ability of metallic post did contribute to the polymerization of dual-cured resin cements at the apical root level. And thickness of resin cements varied from the expected estimated diameter of the prepared space for post cementation.

At the coronal level of canal orifice, exposed marginal areas of resin cements can largely be assisted by light-curing modes; thus ample numbers of photons were available for absorption by the photo-sensitizers, which could help more free radicals formation to initiate and propagate the polymerization process for dual-cured resin cements. Thus, PF group showed higher hardness value at coronal root level. Light transmission properties of cast metal post had been evaluated and it was found that the cast metal post did not allow light transmission in the apical and central portions of the crown (Michalakis *et al*., 2004). According to a previous report significantly lower amount of light reached the apical portion of the root regardless of the type of post was used (Goracci *et al*., 2007). Another finding, in agreement with previous report supports the concern that light intensity at the deepest level of the root canal may be insufficient to induce proper polymerization of adhesive cement (dos Santos Alves Morgan *et al*., 2008). The hardness result of dual-cured resin cement of the present study thus indicates that although the two forms of activation of the curing reaction are present in dual-cured mode, they are supplementary and independent and thus apical portion of the root cement was softer compared to the coronal area. As reduced degree of conversion may lower the mechanical properties of resin cements application and properties of dual cured resin cement with metal post (titanium post) needs further evaluation. As no significant differences on hardness of self-cured group was observed it can be assumed that the polymerization of resin cement from coronal to apical part was adequate as the mode of polymerization was exclusively self-cured type.

In the present study, the cement layer thickness was not significantly different at different levels of each group of cements; and at the same level in between the two groups of cements. There was tendency to become thinner cement at increasing root canal depth regardless of type of cement. The similar cement thickness was probably due to root canal morphologies of upper central incisor that is straight and for the same diameter was maintained for post space preparation for all specimens. It was reported that polymerization stress is an important factor in the process of failure between an adhesive system and crown and root dentin and reported that the thinner the cement layer, the less likelihood of microporosities and polymerization shrinkage (Valandro *et al.,* 2005). Although post space was prepared in such a way so that cement space would be 250 µm, an overall increased in cement layer thickness at all three root canal levels was observed. The increased cement thickness was probably due to manual lateral forces generated during the use of post drill. However, voids and bubbles were present more in PF group, probably due to their viscosity that restricts their placement inside the post space and the mixing procedure of the two types paste system. It is important to evaluate the relationship of polymerization of resin cement and uniformity of cement adaptation with root canal wall as voids and bubbles were more obvious in dual-cured type. The application of resin cements with a lentulospiral instrument was used in other previous in vitro studies, even if not recommended by the manufacturer, is reported as an effective technique to reduce voids and bubbles within the luting agent (Akgungor and Akkayan, 2006). Since applying the cement to the post space with a lentulospiral instrument could result in early set of the apical portion of the cement once it becomes anaerobic, manufacturer's instructions were followed and use of lentulospiral instrument was avoided.

Conclusion

While selecting resin cement for post cementation, the combinations of metallic post and dual-cured resin have to be carefully considered. The hardness results of this study suggest that polymerization of dual-cured resin cement was significantly inadequate at apical root canal level when used with titanium post while thickness of resin cements showed no significant difference at different root levels of both groups. However, the overall increased thickness of resin cements in comparison to post space size needs to be further evaluated. Research on polymerization properties of resin cements with different activation modes in relation to bond strength of metallic/ non-metallic posts will be interesting to evaluate.

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