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Degree of conversion of dual-cured resin cement light-cured through three fibre posts within human root canals: an *ex vivo* study

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Abstract

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Aim To evaluate the degree of conversion of one dual-cured resin cement light-cured through three fibre posts within extracted human teeth.

Methodology Fifteen mandibular premolars were root filled and then divided into three groups. Variolink II was light-cured through the posts (LP, D.T. Light-Post; PP, FRC Postec Plus; SP, Snowpost) within the root canal. The degree of conversion was obtained at 1 mm intervals in 9 mm-deep longitudinally sectioned root canals using an optical microscope connected to an FTIR spectrophotometer (n = 10). The light transmission of each post tested was also examined using UV–Vis spectroscopy. Data were analysed using ANOVA and Tukey's test ($\alpha = 0.05$). **Results** The LP and PP posts revealed a light transmission of 10.2% and 7.7%, respectively, whereas the SP post exhibited a significantly lower value of 0.5%. The degree of conversion mean value ranged from 32.78% to 69.73% depending on the depth and type of post. For all the groups, there were significant decreases in the degree of conversion values for the middle region when compared with those for the cervical region (P < 0.05). Except at a depth of 1 mm, the SP group consistently exhibited significantly lower degree of conversion values than the other groups (P < 0.05). The linear regression analysis revealed a strong correlation between the light transmission of the posts and the overall degree of conversion value for each group ($R^2 = 0.9888$).

Conclusions The decrease in the degree of conversion for Variolink II relative to the depth was dependent on the light transmission capacity of the posts tested.

Keywords: degree of conversion, dual-cured resin cement, fibre post, light transmission.

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Introduction

The use of fibre posts has become popular for the restoration of root filled teeth, mainly due to their good aesthetics and similar elastic modulus to dentine (Pierrisnard *et al.* 2002, Plotino *et al.* 2007). Although fibre post retention is derived more from friction than

true adhesion to the intraradicular dentine (Goracci *et al.* 2005), the posts are usually cemented by a resin cement in combination with an adhesive system. However, the performance of an adhesive technique in root canals can be compromised owing to the characteristics of the root dentine (Ferrari *et al.* 2000, Tay *et al.* 2005). In particular, an adhesive technique using a light-cured resin material can be less than ideal, due to the significantly reduced light intensity in a root canal as a result of light scattering within the resin cement and shadowing produced by both the tooth structure and the post (Musanje & Darvell 2006, Faria e Silva *et al.* 2007).

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Therefore, dual-cured resin cements and adhesive systems combining self-cured and light-cured capabilities have been employed to bond fibre posts in a root canal. Nonetheless, although the two initiation systems allow use with limited light penetration, some dualcured materials have still been reported to be primarily dependent on light-activation (Roberts *et al.* 2004, Teixeira *et al.* 2006). Therefore, sufficient light transmission through the posts is still mandatory, even when dual-cured resin cements are used for post fixation.

Unlike opaque posts, translucent glass or quartz fibre posts seem to transmit light to the internal area of a root canal and induce polymerization of lightcured or dual-cured materials, resulting in an increased cure depth (Lui 1994, Yoldas & Alaçam 2005). However, even translucent fibre posts can limit light transmission to values lower than 40% of incident light and may not guarantee an adequate degree of conversion of the resin materials (Teixeira et al. 2006). Several studies have already presented the degree of conversion values of resin cements light-cured through fibre posts in a root canal. Yet, most of these studies employed microhardness tests to evaluate the degree of conversion for resin cements cured through light-transmittable fibre posts, rather than Fourier transform infrared (FTIR) spectroscopy or Raman spectroscopy, which are more accurate methods for evaluating the value (DeWald & Ferracane 1987, Rueggeberg & Craig 1988). For example, when using a Knoop hardness (KH) test, Roberts et al. (2004) observed that the KH values for a resin composite decreased with the depth in a root canal restored with a fibre post.

Meanwhile, Faria e Silva *et al.* (2007) evaluated the degree of conversion of a dual-cured resin cement used to lute fibre posts with different translucencies. In their

study, which used polyvinylsiloxane molds to simulate root canals and Raman spectroscopy, it was found that the degree of conversion values were dependent on the depth of the root canal and type of post. However, there have been relatively few studies on the degree of conversion of resin cements light-cured within the root canal.

Accordingly, this study evaluated the degree of conversion of a dual-cured resin cement light-cured through three different fibre posts in extracted human teeth using FTIR spectroscopy. Prior to the test, the light transmission capacity of each fibre post was measured using UV–Vis spectroscopy to observe the influence of the post on the degree of conversion.

Materials and methods

This study employed two types of translucent fibre post (LP, D.T. Light-Post, Bisco, Schaumburg, IL, USA; PP, FRC Postec Plus, Ivoclar Vivadent, Schaan, Liechtenstein) and one opaque fibre post (SP, Snowpost, Carbotech, Ganges, France), while Variolink II (Ivoclar Vivadent) was tested as the dual-cured resin cement to measure the degree of conversion. The materials used in the present study are described in Table 1.

The light transmission for each fibre post was measured using a UV–Vis spectrophotometer (UV-1650pc, Shimadzu, Kyoto, Japan). To obtain the reference spectrum, the light beam was passed through a glass slide to which adhesive paper tape containing a rectangular aperture (10 mm × 1 mm) had been fixed, and then through another glass slide. The posts were then individually sandwiched between the two slides, and a transmission spectrum within a range of 400–600 nm was acquired for each post. This test was repeated ten times (n = 10) for each post.

Table 1 Fibre posts and dual-cured resin cemen	t used in this study
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Material (post code)	Manufacturer	Composition	Batch number (post size)
D.T. Light-Post (LP)	Bisco Inc., Schaumburg, IL, USA	Quartz fibre (60 vol%), epoxy resin (40 vol%)	0700001321 (#2)
FRC Postec Plus (PP)	Ivoclar Vivadent, Schaan, Liechtenstein	Glass fibre (61.5 wt%), UDMA, TEGDMA, ytterbium trifluoride, silicon dioxide	K13202 (#1)
Snowpost (SP)	Carbotech, Ganges, France	Silica-zirconium glass fibre (60 vol%), epoxy resin (40 vol%)	531642 (#14)
Variolink II	Ivoclar Vivadent, Schaan, Liechtenstein	Bis-GMA, UDMA, TEGDMA, barium glass filler, ytterbium trifluoride, mixed oxides, Ba-Al-fluoro-silicate glass, catalysts and stabilizers	Transparent Base: J27183 Transparent Low Viscosity Catalyst: H01560

UDMA, uretane dimethacrylate; TEGDMA, triethyleneglycol dimethacrylate; Bis-GMA, bisphenol A diglycidylmetahcrylate.

To take account of the differences in diameter of the posts tested, the light transmission (%) for each post was calculated from the measured value. The absorbance (A) was calculated from the measured transmission data (%T) based on the Beer–Lambert Law (Watts & Cash 1994) as:

$$A = \log_{10} 100 / \% T = 2 - \log_{10} \% T$$

A was then divided by the diameter (path length) of each post (b), and A' obtained as follows:

$$A' = A/b$$

Thereafter, A' was converted again into the light transmission (%) of each post using the following equation:

Light transmission(%) =
$$10^{2-A}$$

The light transmission value (%) at 470 nm, which is the approximate peak wavelength of camphorquinone (CQ) activated by blue light in a light-cured resin material, for each post was employed to compare the light transmission capacity of the posts tested (Neumann *et al.* 2005, Teixeira *et al.* 2006).

Fifteen extracted human mandibular premolars were collected after receiving the patients' informed consent. The teeth were disinfected in 0.5% chloramine, stored in distilled water (Reis et al. 2004), and used within 2 months after extraction. The crowns were removed to obtain a 14-mm-length root. For the root canal treatment, a crown down technique was used with K3 nickel-titanium rotary instruments (SybronEndo, Orange, CA, USA); all the canals were enlarged up to an apical file size of 40, 0.06 taper. All the enlargement procedures were followed by irrigation with 2.5% sodium hypochlorite (NaOCl). The prepared canals were then filled with gutta-percha using a continuous wave compaction technique (E&Q plus, Meta Dental, Elmhurst, NY, USA) and AH26 sealer (Dentsply DeTrey, Konstanz, Germany). The filled roots were stored in relative humidity for 72 h to allow the sealer to set.

After the storage period, the gutta-percha was removed from the root canals using a warm endodontic plugger. The teeth were then randomly distributed into three groups (LP, PP, and SP groups). A 9-mm-deep post space was prepared within the root canal using the corresponding drill system for each post manufacturer. To allow an approximately 1-mm-thick cement space, the post spaces were intentionally overenlarged (Faria e Silva *et al.* 2007). All the posts were cut to a total length of 13 mm, and placed in the prepared root canals. Prior to the cementation procedure, the position of the posts within the root canals was verified using a radiograph to allow uniform thickness of the resin cement around the post.

The mixed paste of Variolink II was inserted into the root canal, and then light-cured for 40 s by placing the tip of the light-curing unit (VIP Junior, Bisco Inc.) on the top of the exposed post so that the light could transmit through the longitudinal axis of the post. The output intensity of 600 mW cm^{-2} was constantly measured during the experiment by a dental radiometer (Model 100, Demetron Research Corp., Danbury, CT, USA). Thereafter, the specimens were stored in distilled water at 37 °C.

After 24 h of storage, the specimens were sectioned longitudinally in a buccolingual direction using a lowspeed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) (Goracci et al. 2005, Faria e Silva et al. 2007). Each sectioned surface was then treated with 37% phosphoric acid for 10 s, 2% NaOCl for 1 min, rinsed with water, and dried with air. The degree of conversion for each post group was measured using a FTIR spectrophotometer (IRPrestige-21; Shimadzu, Kyoto, Japan) connected to a microscope (AIM-8800, Shimadzu) (n = 10). At each observation depth, the centre of the cement layer was designated as the measurement location under the microscope. The measurements were performed at depth intervals of 1 mm using a reflectance mode. The absorbance spectrum was acquired by scanning the specimens 10 times over a $1670-1550 \text{ cm}^{-1}$ range with a resolution of 4 cm^{-1} . The degree of conversion was determined from the aliphatic C=C peak at 1638 cm^{-1} , while the aromatic C=C peak at 1608 cm^{-1} was used as the internal calibration for calculating the final value. The degree of conversion was then calculated by comparing the heights of the peaks for the methacrylate vinyl group in the cured resin with that in the uncured resin, according to the following formula:

Degree of conversion (%) = $(1 - C/U) \times 100$

where C and U are the absorption peaks for the cured and uncured resins, respectively.

As all the data were normally distributed (Kolmogorov–Smirnov test), and the groups exhibited a homogeneous variance (Levene test), a one-way or two-way ANOVA and Tukey's *post hoc* test were employed for the statistical evaluation of the data ($\alpha = 0.05$). Plus, a linear regression analysis was performed to examine the existence of any correlation between the light transmission and the degree of conversion. All the statistical analyses were performed using spss 14.0 for Windows (SPSS Inc., Chicago, IL, USA).

Results

The light transmission for the three fibre posts tested is shown in Fig. 1. The two translucent fibre posts (LP and PP posts) had a mean light transmission capacity of 10.2% and 7.7%, respectively, at 470 nm, representing a significant difference from each other (P < 0.001). Meanwhile, the opaque SP post had a light transmission capacity of less than 1% for all the wavelengths tested, and exhibited a significantly lower value (0.5%) at 470 nm than the two translucent fibre posts (P < 0.001).

Table 2 presents the degree of conversion values for each group at each root canal depth. The mean value ranged widely between 32.78% and 69.73% depending on the depth and type of post; a gradual degree of

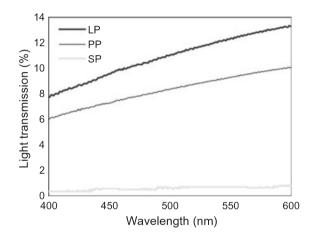


Figure 1 Light transmission (%) of three fibre posts tested. LP, D.T. Light-Post; PP, FRC Postec Plus; SP, Snowpost.

conversion decrease was observed in relation to the depth for all the groups. The two-way ANOVA revealed that the depth (P < 0.001), post (P < 0.001), and interaction between these two factors (P = 0.007) all had a significant affect on the degree of conversion. Thus, the degree of conversion decrease according to the depth was found to be post-dependent.

For all the groups, there were significant decreases in the degree of conversion values for the middle region when compared with those for the cervical region (P < 0.05), although significant degree of conversion decreases were detected within the cervical region for the PP and SP groups (P < 0.05). In the middle region, significant degree of conversion decreases was also observed depending on the type of post (P < 0.05). Within the apical region, the degree of conversion values for each group was statistically similar to each other, irrespective of the depth (P < 0.05). Except at a depth of 1 mm, where no significant differences in the degrees of conversion were found among the groups (P = 0.262), the SP group consistently exhibited significantly inferior degree of conversion values than the other two groups (P < 0.05).

The overall degree of conversion values, which were pooled together for each group regardless of the depth, are summarized in Table 3. According to the Tukey's *post hoc* test, there was no significant difference in the degree of conversion between the LP and PP groups (P = 0.065), while the SP group exhibited a significantly lower degree of conversion value than the other two groups (P < 0.001).

Figure 2 depicts the linear regression analysis results, which revealed a strong correlation between the light transmission capacity of the posts tested and

Region	Depth (mm)	D.T. Light-Post (LP)		FRC Postec Plus (PP)		Snowpost (SP)	
		Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
Cervical	1	69.73 Aa	5.72	68.68 Aa	5.98	65.86 Aa	4.67
	2	69.24 Aa	3.89	67.07 Aa	2.84	58.84 Bb	3.25
	3	64.58 Aa	2.20	60.56 Ba	4.66	53.09 Bb	6.88
Middle	4	56.91 Ba	4.14	53.93 Ca	3.02	45.45 Cb	4.76
	5	53.72 BCa	3.68	47.05 Db	3.66	40.26 CDc	4.05
	6	50.17 CDa	5.26	42.37 Db	5.06	34.69 DEc	4.22
Apical	7	45.91 Da	3.13	44.14 Da	4.71	34.04 Eb	2.00
	8	46.84 Da	4.36	42.09 Db	3.17	32.78 Ec	2.32
	9	46.50 Da	3.63	44.07 Da	2.69	32.89 Eb	3.39

 Table 2 Degree of conversion (%) (mean and standard deviation) of Variolink II light-cured through three fibre posts tested at different depths

Mean values followed by different upper-case letters and different lower-case letters differed within the column and row, respectively, according to the Tukey's post hoc test (P < 0.05).

Table 3 Overall degree of conversion (%) (mean and standard deviation) for each group

D.T. Light-Post (LP)		FRC Postec Plus (PP)		Snowpost (SP)	
Mean	Standard deviation	Mean	Standard deviation	Mean	Standard deviation
55.95 a	9.98	52.22 a	10.91	44.21 b	12.42

Values are mean and standard deviation obtained by pooling all data from the same group. Mean values followed by different letters differed according to the Tukey's *post hoc* test (P < 0.05).

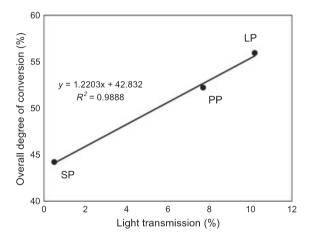


Figure 2 Linear regression analysis showing correlation between light transmission of three posts tested and overall degree of conversion for each group. LP, D.T. Light-Post; PP, FRC Postec Plus; SP, Snowpost.

the overall degree of conversion value for each group $(R^2 = 0.9888)$.

Discussion

This study evaluated the change in the degree of conversion of Variolink II, a dual-cured resin cement, light-cured through three different posts measured at 1-mm intervals in 9-mm-deep root canals with the help of an optical microscope connected to an FTIR spectrophotometer. In a previous Raman spectroscopic study by Faria e Silva *et al.* (2007), they used a rubber impression material to simulate root canals for degree of conversion measurements of a dual-cured resin cement in a near-clinical situation. Although the dual-cured resin cement and fibre posts differed between the two studies, the degree of conversion mean values obtained in the current study, ranging from 32.78% to 69.73% depending on the depth of the root canal and

type of post (Table 2), were similar to those in the previous study (29.00–69.80%). Plus, the degree of conversion mean values for the three test groups obtained for the cervical region in this study (53.09–69.73%) were approximately similar to the results of another previous study (Kumbuloglu *et al.* 2004) in which the degree of conversion was approximately 60% when the material was light-cured.

When compared with the cervical region, there were significant decreases in the degree of conversion values for the middle region for all the groups, implying that the polymerization of the dual-cured resin cement Variolink II may still be dependent on light activation. Indeed, the degree of conversion mean values decreased to between 32.78% and 46.84% in the apical region. Therefore, it would seem that the amine in the base paste and peroxide in the catalyst paste were unable to react effectively in the self-curing mode. Several studies have already shown that light activation is still required for some dual-cured resin cements to increase the degree of conversion, even though the self-curing and light-curing modes of activation are independent (Braga et al. 2002, Lu et al. 2005). Thus, the degree of conversion decrease when increasing the distance from the curing tip was probably due to a significant reduction in light intensity within the root canal.

Moreover, the tendency of the degree of conversion to decrease within the root canals seemed to be postdependent, i.e. related to the light transmission capacity of the post tested. As expected, the light transmission of the SP post was extremely low (0.5% at 470 nm) (Fig. 1), due to its opaque nature. Since this post contains a high amount of silica-zirconium glass fibres embedded in an epoxy resin matrix (Table 1), it is not intended to allow the transmission of light. Therefore, another version (Snowlight, Carbotech) presented by the same manufacturer are also available to improve the light transmission. Meanwhile, although the quartz fibre LP post and glass fibre PP post showed a significantly higher light transmission capacity than the SP post, the mean values (10.2% for the LP post and 7.7% for the PP post at 470 nm) still seemed to be relatively low. In an assessment of fibre post systems by Teixeira et al. (2006), the LP post exhibited a higher light transmission at 470 nm (22%) when compared with the value in the current study (10.2%). This inconsistency may have been attributable to the large variance in the evaluation methods of the light transmission behaviour.

Thus, the intensity of the light transmitted to the resin cement within the root canal may be attenuated depending on the distance from the curing tip and the light transmission capacity of the post. In the cervical region for the LP group, there were no significant differences in the degree of conversion values, implying that the light from the light-curing unit effectively activated the CQ in the resin through the LP post at this depth. In contrast, for the PP and SP groups, significant degree of conversion decreases were found within the region, seemingly due to the reduced light transmission of the posts and restricted self-curing capacity of the resin cement. The degree of conversion values for all the groups significantly decreased for the middle region when compared with those for the cervical region, probably due to a substantial reduction in the light intensity, however, the LP group still exhibited similar or significantly higher degrees of conversion than the other two groups at this depth.

In the apical region, the LP group consistently showed similar or significantly higher degrees of conversion than the other two groups, while the SP group showed significantly lower degrees of conversion. These phenomena may be partly explained by the relative superior light transmission capacity of the LP post, i.e. the post was able to transmit light to a deeper depth to activate the light-cured component of the polymerization system. Thus, although the polymerization reaction in the apical third would be expected to rely mainly on the self-curing mode, the significantly higher degree of conversion for the LP and PP groups compared to that for the SP group would seem to imply that the resin cement was also light-cured to some extent even at this depth.

When considering the overall degree of conversion values, the LP group exhibited a superior mean value (55.95%), followed by the PP and SP groups (52.22% and 44.21%, respectively) (Table 3). However, the statistical analysis did not reveal any significant differentiation between the LP and PP groups based on a large variance (standard deviation). Nonetheless, the linear regression analysis showed a strong correlation between the light transmission capacity of the posts tested and the overall degree of conversion value for each group (Fig. 2), suggesting that the light transmission capacity of a fibre post would appear to be a critical factor determining the degree of conversion of dualcured resin cements polymerized within the root canals.

Although this study attempted to mimic a clinical situation using extracted human teeth, certain limitations to the experimental design were present. Above all, the microscopic technique employed in this study required a bigger aperture analysis area from a realtime image to obtain stable FTIR spectra. In the present study, the resin cement thickness was approximately 1 mm, which was almost uniform throughout the root canals. However, since this is considerably thicker than that generally found in fibre post cementation (Grandini et al. 2005), a thinner resin cement thickness may vary the resultant degree of conversion values to some extent. In addition, although fibre post systems with a similar diameter, taper, and shape were selected for this study, a slightly different post configuration could affect both the light transmission and the degree of conversion results (Lui 1994). Cylindrical fibre posts could present more reliable results than the tapered fibre posts, even though the influence might be minimal. In addition, only one dual-cured resin cement and three fibre posts products were tested in the present study. Thus, other dual-cured resin cements could produce different results depending on the comparative selfcured and light-cured capabilities and light transmission of the post. Finally, the influence of periodontal tissue on the degree of conversion value of the dualcured resin cement was not simulated. Nonetheless, despite such limitations, the degree of conversion values obtained in this study can still be utilized to estimate the value of resin cements used for fibre post luting in a clinical situation.

A poorly-cured resin cement within the root canal, especially at the apical portions, may sometimes jeopardize post retention and threaten the survival of the restoration and tooth (Giachetti et al. 2004). Hayashi et al. (2008) reported that self-cured totaletch adhesive materials may have an advantage in post-core restorations, probably due to stable bonding performances in an entire post space. However, care must be taken not to attribute clinical success to degree of conversion values alone, as these values are not the only factor determining clinical performance (Lu et al. 2005, Faria e Silva et al. 2007). Although imperfect polymerization of the light-cured materials at the apical portions may produce the inferior bond strengths (Hayashi et al. 2008), a major contributor to the post fixation strength seems to be the interfacial sliding friction (Goracci et al. 2005). Therefore, the degree of conversion values measured in this study does not necessarily translate into post fixation behaviour, i.e. the bond strength of the fibre post to the root dentine. Nonetheless, adequate polymerization of resin cements is also desirable to improve a material's biocompatibility by reducing the amount of residual monomers leached into the oral environment (Carmichael et al.

1997, Hansel *et al.* 1998). The leachable monomers from a poorly-cured resin cement can leak through the apical foramen and induce some detrimental effects in the periodontal tissue (Janke *et al.* 2003, Souza *et al.* 2006).

Thus, within the limitations of this study, fibre posts with a superior light transmission capacity and dualcured resin cements with a higher self-cure activity would appear to be preferable for fibre-post cementation. Moreover, even when a dual-cured resin cement is used for luting a fibre post, a longer or extended curing time would be recommended to improve the maximum degree of conversion (Lu *et al.* 2005). Yet, further research is still needed to clarify the polymerization of dual-cured resin cements used for fibre-post luting.

Conclusion

Within the root canal, the decrease in the degree of conversion for Variolink II light-cured through three fibre posts, ranging from 32.78% to 69.73%, was found to be dependent on the depth and type of post. In addition, the linear regression analysis revealed a strong correlation between the light transmission capacity of the posts tested and the overall degree of conversion value for each group.

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