

Influence of post type on degree of conversion of a resin-based luting agent

CHIARA OTTAVIA NAVARRA, DDS, PhD, CECILIA GORACCI, DDS, PhD, LORENZO BRESCHI, DDS, PhD, ALESSANDRO VICHI, DDS, PhD, GABRIELE CORCIOLANI, DDS, PhD, MILENA CADENARO, DDS, PhD & MARCO FERRARI, MD, DDS, PhD

ABSTRACT: Purpose: To assess the degree of conversion (DC) at different levels of a resin-based luting agent used with different commercial posts. **Methods:** A resin-based luting agent (Variolink II) was light-cured in a simulated dowel space in combination with different fiber posts: GC Fiber Post (GC), DT Light Safety Lock (SL), RelyX Fiber Post (RX), Reforpost (RE), DT Light Post Illusion (LPI), FRC Postec Plus (FP), Radix (RA), Snowpost (SP), Dentin Post X (DP), Macrolock Illusion (MI), and Composipost (CP). After 24 hours, DC was assessed through micro-Raman analysis at the coronal, middle, and apical thirds. Two-way ANOVA and Tukey test were used for post-hoc comparisons ($P < 0.05$). **Results:** The results demonstrated that the post-type, dowel space level, and between-factor interaction were statistically significant ($P < 0.001$). Differences in DC were observed at different depths for RX, RE, LPI, SL, DP, MI and CP, decreasing from the coronal to the apical third. Although the resin-based luting agent should be able to polymerize in absence of light, a higher DC was found following light irradiation (with a decreasing effect from the coronal to the apical third). (*Am J Dent* 2012;25:17-20).

CLINICAL SIGNIFICANCE: Fiber post type influenced the degree of conversion of the tested resin-based luting agent.

✉: Prof. Milena Cadenaro, Department of Medical Sciences, University of Trieste, Piazza Ospedale 1, 34129 Trieste, Italy. E-mail: m.cadenaro@fmc.units.it

Introduction

Advances in fiber-reinforced composite posts have led to a substantial increase in their clinical use in the last 20 years.^{1,2} The restoration of endodontically treated teeth with fiber posts has overcome the majority of the limitations of metallic posts (esthetic appearance, mode of failure, clinical performance),³⁻⁵ and translucent glass fiber posts are frequently preferred to carbon fiber posts for esthetic reasons.

The retention of post-and-core restorations depends both on the quality of the bond, and the mechanical properties of the material at the different interfaces: intraradicular dentin–adhesive, adhesive-luting agent, and luting agent–fiber posts. Previous clinical^{6,7} and laboratory^{8,9} studies have evaluated the retention of fiber posts, particularly at the dentin interface, both at the coronal and intraradicular level.¹⁰ To allow optimal sealing, retention, and favorable stress distribution inside the root canal, resin-based luting agents were indicated for fiber post cementation.^{8,9} Resin-based luting agents (RBLA) have higher mechanical properties and increased retentive and sealing capabilities in comparison with conventional luting materials, such as zinc phosphate-based¹¹ and glass-ionomer cements.¹² Currently, the majority of the resin-based luting agents are used in combination with etch-and-rinse or self-etch adhesive dentin bonding agents. Self-adhesive or “self-adhering” (RBLA) have been introduced to simplify the luting procedure and to reduce operator-related technique sensitivity that may compromise bonding effectiveness.¹³

RBLA are available as light-, self-, or dual-cure formulations. To ensure optimal mechanical properties, correct polymerization should be provided both at the coronal and apical level,^{14,15} as a low degree of conversion (i.e., converted double bond percentage, DC) compromises the mechanical properties.¹⁶ Adequate curing is particularly important in the apical portions of the root canals, because impaired adhesion has been demonstrated in a large number of studies analyzing the adhesion along the post space.⁸

A previous investigation¹⁷ revealed the existence of variability in the light transmission among the several types of posts available on the market. Indeed, when translucent posts are used, polymerization can be jeopardized in the deepest areas of the canal space due to limited light transmission.¹⁸ Consequently, the use of self- and dual-cure RBLA has been advised as a safe procedure in post luting, to optimize the final DC of the RBLA.¹⁹ Dual- and self-cure RBLA are expected to properly polymerize in areas that cannot be entirely reached by light, or in the complete absence of light. Nevertheless, because suboptimal polymerization of different dual-cured RBLA was reported in the absence of light,^{20,21} light curing is usually recommended even for dual-cured RBLA.

To investigate the polymerization within the small simulated post space, micro-Raman microscopy was used. In fact, this technique can be used to identify the chemical distribution of polymers within the post-luting agent interface and to accurately determine the DC by measuring the amount of converted double bonds comparing the tested material in the uncured and cured states.²²⁻²⁴ Additionally, the technique allows minimal sample manipulation and in situ monitoring of the substrate, resulting in a spatially resolved chemical analysis, along with a non-destructive technique that is unaffected by sample thickness.²⁴

This study assessed the degree of conversion achieved at different levels of a simulated dowel space by a dual-cure RBLA used in combination with different types of fiber posts. The first tested null hypothesis was that the post-type would not significantly influence the DC of the RBLA. The second tested null hypothesis was that differences in RBLA DC would not be observed at different depths along the RBLA/post interface.

Materials and Methods

Specimen preparation - Posts of different shape and composition were selected (Table 1). To standardize the geometric properties, the post-sizes that were most similar among the different brands were selected (Table 1). Additionally, to stan-

Table 1. Composition and characteristics of the tested fiber posts.

Post	Abbreviation	Size	Fibers	Resin matrix	Shape
GC Fiber Post	GC	1,2	Glass	Methacrylate	Double-tapered
DT Light Safety Lock	SL	1	Pre-conditioned quartz	Epoxy	Double-tapered
RelyX Fiber Post	RX	1	Glass	Resin	Double-tapered
Reforpost	RE	1	Glass	Bis-GMA	Serrated
DT Light Post Illusion	LPI	1	Quartz	Epoxy	Double-tapered
FRC Postec Plus	FP	1	Glass	UDMA, TEGDMA, ytterbium trifluoride, highly dispersed silicon dioxide	Tapered
Radix	RA	1	Zirconium enriched glass	Epoxy	Double-tapered
Snowpost	SP	1	Zirconia-rich glass	Epoxy	Cylindrical with long apical cone
Dentin Post X	DP	444L12.070	Glass	Epoxy	Tapered with a retentive head
Macrolock Illusion	MI	1	Quartz	Epoxy	Tapered, circumferential head grooves, spiral head serrations
Composipost	CP	1	Carbon	Epoxy	Two-stage parallel

Table 2. Composition of the resin based cement and the mode of application recommended by the manufacturer.

Cement	Composition (wt%)	Application mode
Variolink II - Base	Dimethacrylates (26.3) inorganic fillers (silica, barium glass, ytterbium trifluoride) (73.4), catalyst, stabilizers (0.3), pigments (<0.1)	1. Apply the adhesive. 2. Mix Variolink II in a 1:1 ratio for 10 seconds. 3. Seat post. 4. Polymerize at least 40 seconds
Catalyst	Dimethacrylates (27.9) inorganic fillers (silica, barium glass, ytterbium trifluoride) (71.2), catalyst, stabilizers (0.9), pigments (<0.1)	

dardize the length, each post was cut at 15 mm from the tip using a diamond bur (FG 4205 L^j), mounted on a high-speed hand piece (Super-Torque Lux 3 650B^k) under a water spray. A metal guide was used to position the post, centered within a cylindrical metal mold, simulating the dowel space of 13 mm height, 3 mm diameter, and filled with a luting agent.

The tested RBLA was Variolink II,^f a two component self-curing with redox-initiated polymerization. The formulation and application mode are described in Table 2. Variolink II was light-cured (L.E. Demetron 1^l). The irradiance at the tip of the curing light was 700 mW/cm² as previously assayed by a LED radiometer (100 Optilux^m radiometer) for 20 seconds, placing the light tip on the coronal extremity of the post. After 24 hours in air storage, cured specimens were cut along the post axis using a low-speed diamond saw under water-cooling (Micro-metⁿ) to expose the post/RBLA interface and prepared for the micro-Raman analysis.²³

Real-time Raman spectroscopy - The Raman spectrometer was calibrated before each experiment using an integrated software (WiRE 2.0^o) with a silicon single crystal with a band of 520 cm⁻¹, following the manufacturer's specifications.

Variolink II was dispensed into a pan under dark conditions. Real-time Raman spectra of the RBLA were collected through a Raman microprobe in the back-scattered configuration, equipped with a Leica DM/LMP optical microscope with a x20 objective (Leica N Plan EPI objective^p). A 785-nm wavelength laser (150 mW) was used to collect the spectra (Renishaw

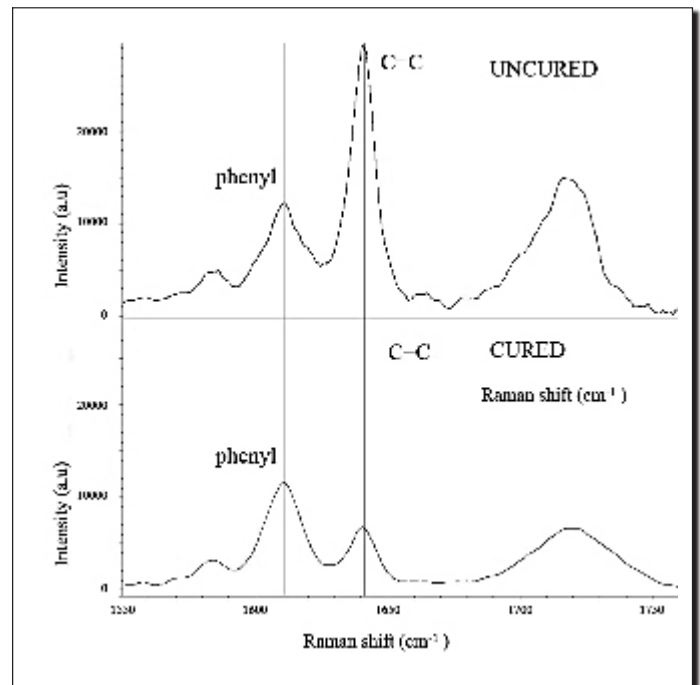


Figure. Raman spectra of the cured and uncured RBLA. The peaks used to calculate the degree of conversion are detectable. The 1610 cm⁻¹ (phenyl group) was chosen as a reference peak and the 1640 cm⁻¹ (C=C double bond) as a reaction peak. The decrease of the C=C peak after polymerization is visible when comparing the spectra of the uncured RBLA (first spectrum on the top) and of the cured RBLA (at the bottom).

InVia^o). The spectral coverage of this model ranged from 100 to 3450 cm⁻¹ with an average spectral resolution of 5 cm⁻¹. Spectra centered at 1640 cm⁻¹ were continuously collected with an exposure time of 0.1 seconds. Since the RBLA without initiators could not be provided by the manufacturer, a representative spectrum was recorded in the first 30 seconds of the self-polymerization reaction, when no significant changes in the C=C peak intensity and area was found, and used as the uncured reference at baseline.

Micro-Raman spectroscopy - Post slabs with exposed luted interfaces were placed directly on the calcium fluoride microscope slide and spectra of the bonded interface acquired at 1 mm (coronal, C) and 4 mm from the coronal margin (middle, M), and at 0.5 mm from the apex (apical, A). The

Table 3. Degree of conversion expressed as mean DC% (SD) of Variolink II cement with the tested posts. Different capital superscript letters label statistically significant differences among post- types, regardless of the dowel space level. Different superscript capital letters label statistically significant differences among the dowel space levels, regardless of the post-type. Different small superscript letters label statistically significant differences among the post-types within each post space level (vertical columns). Different symbols label statistically significant differences among the post space levels within each post type (vertical columns).

Level	Post										
	GC ^A	SL ^{AB}	RX ^{AB}	RE ^{AB}	LPI ^{AB}	FP ^{AB}	RA ^{AB}	SP ^{ABC}	DP ^{ABC}	MI ^{BC}	CP ^C
Ca	76 ^{ab} (3)	81 ^{a*} (2)	76 ^{ab*} (6)	78 ^{ab*} (6)	77 ^{ab*} (2)	71 ^b (1)	73 ^{ab} (4)	75 ^{ab*} (2)	73 ^{ab*} (2)	76 ^{ab*} (0)	75 ^{ab*} (2)
Mβ	72 ^{ab} (6)	73 ^{ab§} (4)	76 ^{a*} (4)	74 ^{ab*} (2)	69 ^{ab§} (0)	73 ^{ab} (3)	69 ^{ab} (5)	65 ^{b§} (4)	71 ^{ab*} (2)	72 ^{ab*} (0)	72 ^{ab*} (2)
Aγ	74 ^{ab} (4)	67 ^{abc#} (6)	67 ^{abc§} (1)	65 ^{bcd§} (2)	71 ^{abc*§} (0)	70 ^{abc} (6)	69 ^{abc} (2)	70 ^{abc*§} (1)	64 ^{bcd§} (1)	58 ^{bcd§} (0)	47 ^{e§} (2)

Abbreviations: GC: GC Fiber Post; SL: DT Light Safety Lock; RX: RelyX Fiber Post; RE: Reforpost; LPI: DT Light Post Illusion; FP: FRC Postec Plus; RA: Radix; SP: Snowpost; DP: Dentin Post X; MI: Macrolock Illusion; CP: Composipost.
C: coronal; M: middle; A: apical.

spectral region included 400 cm⁻¹ to 1900 cm⁻¹, using a x100 objective (Leica N Plan EPI objective), to increase the spatial resolution by a laser spot size of ≤ 1 μm. The 785-nm laser power on the surface was ~8 mW with an exposure time of 40 seconds for each scan. Acquired data were analyzed with spectrographic analysis software (Grams/AI 7.02^q).

Degree of conversion calculation - Raman spectra of both the uncured and cured RBLA were collected to identify the reference and reaction peaks of each material. Spectra were characterized by identifying the typical bands of methacrylate-based materials: 1610 cm⁻¹ (phenyl C=C)^{24,25} and 1640 cm⁻¹ (C=C group).^{22,23} The phenyl 1610 cm⁻¹ peak was selected as a reference peak (it remained stable during polymerization), and the 1640 cm⁻¹ was selected as a reaction peak (Figure). The DC was calculated using the ratio between the reaction (Arxn) and internal reference (Aref) peak areas as follows:

$$\%conversion = \left\{ 1 - \frac{[A_{rxn}(p)/A_{ref}(p)]}{[A_{rxn}(u)/A_{ref}(u)]} \right\} \times 100$$

where “u” refers to uncured system and “p” to cured system.

Statistical analysis - Because the data distribution was normal and group variances were homogeneous, two-way ANOVA was used, with the percentage of DC as the dependent variable, and the post-type and dowel space level as factors. The Tukey test was used for post-hoc comparisons, as required. In all the analyses, the level of significance was P < 0.05.

Results

The mean and standard deviation values of DC percentages are reported in Table 3. Post type, dowel space level, and between-factor interactions were statistically significant (P < 0.001). Specifically, regardless of the post-space level, with GC, the DC of the RBLA was significantly higher than MI and CP (P < 0.05). In the presence of the CP, DC was significantly lower than with any other post apart from MI (P < 0.05). Independently from post type, significantly decreasing DC values were measured from the coronal to the apical third (P < 0.05). Regarding the between-factor interaction at the coronal level, significantly higher DC percentages were measured with SL compared to those with FP (P < 0.05). At the middle level, the DC was significantly higher with RX than with SP (P < 0.05). At the apical level, the highest DC was recorded in the

presence of GC and the difference was statistically significant with MI, DP, and RE (P < 0.05). When using CP, DC was significantly lower than with any other post (P < 0.05). With RX, RE, SL, LPI, CP, DP, and MI, the DC was significantly higher at the coronal and middle levels than apically (P < 0.05). With LPI and SP, DC at the coronal level was significantly higher than at the middle level (P < 0.05).

Discussion

The first research hypothesis was rejected because the post-type did influence the RBLA DC. Despite their claimed translucent properties, significant differences were observed between the different translucent posts tested in the study, with GC leading to a significantly higher DC of the cement, regardless of the post space level. In contrast, the carbon opaque fiber post CP showed lower DC values compared with the translucent posts, particularly at the apical level. The DC obtained with CP at the apical and middle level was similar to that of other translucent posts, suggesting that although light cannot be transmitted through carbon fibers, the translucency of the RBLA itself can allow light passage and reach the initial millimeters of the post space.

The second null hypothesis was also rejected because differences in DC were observed at different depths along the RBLA/fiber post interface for some of the tested posts, decreasing from the coronal to the apical third (RX, RE, LPI, SL, DP, MI, CP) (Table 2). The shape, composition and translucency of fiber posts determined differing light transmission among the different posts and thus could influence the curing reaction of the LA.^{26,27} Large variations in translucency were observed among marketed fiber posts¹⁷ and the post-light transmitting ability was found to have an influence on DC,²⁶ hardness,^{27,28} and elastic modulus²⁷ of LA. Goracci *et al*¹⁷ investigated the translucency of different marketed fiber posts at different depths, finding a wide range of variability among manufacturers, with the constant of a decreased translucency from the coronal to the apical level, and an increase at the post tip. Comparing these results with the present study, no direct correlation was found. GC showed the highest DC even though it was not the most translucent, while RX and DP, which appeared to be the most translucent at the apical level,¹⁷ showed a lower DC compared with the other posts. In contrast, for some posts (GC, FP, RA, SP) a conversion of the RBLA comparable to the coronal level was found in the apical level,

and for FP and SP, the translucency at this level appeared low, according to Goracci *et al.*¹⁷ The variations are likely due to the complex interactions of the photo- and self-polymerization occurring in RBLA that cannot be investigated independently.²⁹ Moreover, Variolink II must be mixed manually before its application; thus, operator-dependent variability in the levels of the components used, or in the mixing time may have led to differences in the final DC.³⁰ An additional factor that may have influenced the DC values recorded in the study is that the Raman analysis was performed 24 hours after specimen preparation. Had the specimens been tested at an earlier stage, higher difference in DCs may have been seen. The DC values obtained in this study are comparable to those recently reported, in which the DC of different RBLA, including Variolink II, was investigated after 24 hours around ceramic inlays using micro-Raman analysis.³¹

In conclusion, the post-type and dowel space depth do influence DC. It would thus be interesting to assess whether the DC obtained at different depths also reflected different post retentive strengths. Correlative push-out measurements may provide useful data to determine this.

- a. GC, Tokyo, Japan.
- b. VDW GmbH, Munich, Germany.
- c. 3M ESPE, St. Paul, MN, USA.
- d. Angelus, Londrina, PR, Brazil.
- e. RDT, St. Egreve, France.
- f. Ivoclar-Vivadent, Schaan, Liechtenstein.
- g. Dentsply International, York, PA, USA.
- h. Abrasive Technology, Inc, Lewis Center, OH, USA.
- i. Komet-Brasseler, Lemgo, Germany.
- j. Intensiv, Grancia, Switzerland.
- k. KaVo Dental, Biberach/Riss, Germany.
- l. Kerr, Orange, CA, USA.
- m. sds Kerr, Danbury, CT, USA.
- n. Remet, Bologna, Italy.
- o. Renishaw plc, Wotton-under-Edge, Gloucestershire, UK.
- p. Leica, Wetzlar, Germany.
- q. Thermo Galactic Inc., Salem, NH, USA.

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Dr. Navarra is post-doctoral Fellow, Dr. Goracci is Assistant Professor, Dr. Breschi is Associate Professor, and Dr. Cadenaro is Associate Professor, Department of Biomedicine, Division of Dental Sciences and Biomaterials, University of Trieste, Trieste, Italy. Dr. Vichi is Research Professor, Dr. Corciolani is Clinical Instructor, and Dr. Ferrari is Professor and Chair, Department of Fixed Prosthodontics and Dental Materials, University of Florence and Siena, Policlinico Le Scotte, Siena, Italy.

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