Degree of conversion of dual-cure resins light-cured through glass-fiber posts

ARTICLE *in* AMERICAN JOURNAL OF DENTISTRY · FEBRUARY 2011

Impact Factor: 0.85 · Source: PubMed

7 AUTHORS, INCLUDING:

Massimo Gagliani
University of Milan
80 PUBLICATIONS **966** CITATIONS
SEE PROFILE

Marco Ferrari
Università degli Studi di Siena
503 PUBLICATIONS **10,249** CITATIONS
SEE PROFILE

Laura E. Depero
Università degli Studi di Brescia
324 PUBLICATIONS **3,548** CITATIONS
SEE PROFILE

Antonio Cerutti
Università degli Studi di Brescia
110 PUBLICATIONS **121** CITATIONS
SEE PROFILE

Research Article

Degree of conversion of dual-cure resins light-cured through glass-fiber posts

FRANCESCA CERUTTI, DMD, **PIER ANTONIO ACQUAVIVA**, DMD, **MASSIMO GAGLIANI**, MD, DDS, **MARCO FERRARI**, MD, DDS, **FRANCESCO MANGANI**, MD, DDS, **LAURA E. DEPERO**, PROF & **ANTONIO CERUTTI**, MD, DDS

ABSTRACT: Purpose: To evaluate the degree of conversion (DC) of dual-curing materials used to lute glass-fiber posts in a simulated root canal polymerized by two different modalities. **Methods:** Artificial root canals were used to simulate a clinical condition to lute 45 posts by three different dual curing luting cements (Calibra, Multilink Automix and Variolink II). Two light cure modalities were chosen for each luting cement: standard (S group) 400 mW/cm^2 for 120 seconds and high-power (H group) 1200 mW/cm^2 for 40 seconds. Raman spectra were collected at different positions in the post surface (1, 3, 5 and 7 mm from the coronal-most portion of the post covered in cement) and the percentage degree of conversion was computed. The data were analyzed using ANOVA and post-hoc Student-Neuman-Keuls *t*-test (P= 0.05). **Results:** The DC of the tested luting composites decreased progressively while increasing the distance from the light tip. Regardless of the polymerization modality (H or S) applied. Conversely, the curing modality significantly influenced the DC of the tested materials, evidencing different responses to the same energy density: Calibra seemed to be less dependent on light-curing than the other tested materials, showing a constant behavior. Multilink Automix reached the highest DCs in the S group compared to the H mode. Variolink II showed an interesting drawback in DC at 7 mm when cured in the H model. Dual-cure materials show adequate monomer conversion but when the distance from the curing light increased, a variable, but significant lowering in conversion rate was observed. In addition, the time and power of curing appeared to be material-dependent and should be calibrated individually. (*Am J Dent* 2011;24:8-12).

CLINICAL SIGNIFICANCE: To obtain the best performances with dual curing luting materials, a careful choice of the polymerization modality should be taken into account and it could be provided by manufacturers.

✉: Dr. Francesca Cerutti, Department of Materials for Engineering, University of Brescia, Italy. E-✉: fc@francescacerutti.it

Introduction

The post-endodontic restoration of a compromised tooth may require the use of an intracanal post,[1] which has proved reliable[2-6] as a solution to retain the restoration core. Nevertheless, it should be considered that the long-term durability of this restorative procedure[5,7,8] is influenced by post luting.

The performance of the adhesive technique in root canals is affected by the highly unfavorable cavity configuration factor, the difficulty of light-curing the adhesive system or the presence of moisture such as blood or saliva (avoided by the use of the rubber dam).[9]

The material mechanical performances are strongly influenced by the degree of conversion (DC)[10,11] of the monomer present in the resin composite matrix, affected by irradiation intensity, time and mode.[12,13]

In the last decades, high-power light curing units were developed in order to reduce the irradiation time, based on the concept that the same DC is produced by a fixed energy amount (energy density: Joule/cm^2), independent of variations in light irradiance.[14,15]

With light-activated systems, DC decreases as the distance from the light-tip surface increases, due to the attenuation of the radiant energy as it passes through the material.[16-18] Studies of penetration/displacement, Vickers/Knoop hardness, and Shore microhardness[19-22] reported that a light-transmitting post within the resin increases the depth of cure. One study[23] on high-power curing units agreed with the previous findings, concluding that for all curing units tested increasing the light tip-composite distance or decreasing the exposure time decreased the depth of cure.

Dual-cured resin-based luting materials include the advantages offered of self- and light-curing components, characterized by extended working time and capability to convert independently from the presence of light.[24] Nevertheless, some dual-cure resins are primarily dependent on light-activation, so an inadequate DC is expected when light from the curing unit is not available.[25] During post fixation, exposed marginal areas can be assisted by both self- and light-curing modes because they are accessible to the curing light. As light is irradiated, a significant reduction in intensity occurs as a result of the light scattering within the cement and shadowing produced by both tooth structure and post.[26]

Studies that used light-cured resin composites reported that light-transmitting fiber posts increased the extension of cure.[19,27] Silva *et al*[28] evaluated the ability of fiber posts of different translucencies to increase the DC of a dual-cured resin composite, finding a drawback in DC at the apical third.

Currently, there are no studies in the literature comparing the DC of different new generation dual-curing materials employed in endodontic post luting.

The Raman spectroscopy analysis, measuring the non-reacted methacrylate groups, is an accurate, non-destructive and fast on-line technique for assessing the DC of luting composites[27,29-31] with spatial resolution of a micron or better.

This laboratory study evaluated the DC of dual-curing materials used to lute glass-fiber posts in a simulated root canal, comparing polymerization carried out according to two different modalities. The null hypothesis was that there would be no difference in the DC between the materials cured with different modalities, both in the upper part of the simulated root canal and in the lower part of the same.

Table 1. Materials tested in this study. Their composition is quite similar in percentages; the shade is comparable.

Material	Monomer	Filler	Color
Calibra	Bis-GMA	Glass particles, silica, boron, fluoride, aluminum, titanium dioxide	Transparent
Multilink Automix	HEMA	Barium glass, ytterbium trifluoride, mixed spheroidal oxides	Transparent
Variolink II	Bis-GMA, urethane dimethacrylate, triethylen glycol dimethacrylate	Barium glass, ytterbium trifluoride, barium silicates aluminum, fluoride, mixed spheroidal oxides	Transparent

Materials and Methods

Set-up of the simulated root canal - Three custom templates made from mirror-finished Böhler M333-Isoplast[a] steel were produced, each consisting of two slides (5.4×5.4×1.4 cm) joined by a precision connection and blocked by a vice. Between the two tightly adherent slides, a 10 mm-long artificial canal was created in which the diameter increased from 0.9 mm at the apex to 1.6 mm at the top. Each slide was finished until it had a perfectly smooth surface, to prevent adhesion of the luting material to the post space walls.

Sample distribution - The study examined 30 composite fiber-reinforced, translucent, radiopaque, 20 mm-long, and 1 mm in apical diameter, 5°18' tapered posts (FRC Postec Plus Size 3[b]). These were split into two groups (H and S, n = 15) according to the polymerization modality applied during the luting procedure.

Light irradiation - The samples were polymerized using a Swiss Master Light[c] halogen lamp (bandwidth from 400 to 510 nm), keeping the light tip orthogonal to the post and at a constant distance from the apex (the original length of the post was maintained and the light tip was positioned over its coronal portion). Samples belonging to Group S were cured at 400 mW/cm^2 for 120 seconds, while those in Group H were cured at 1200 mW/cm^2 for 40 seconds. The amount of energy provided to each sample was 48 J. The light-curing unit employed in this study was new and, before luting and after every test series, its output was checked by means of its own radiometer.

After 6 minutes, as indicated by the manufacturers, to permit the luting composite setting, the template was opened, the samples were extracted from the artificial canal and stored in a sealed box at 37°C for 24 hours to allow the completion of the chemically activated reaction.

Luting cements - According to previous experimental data,[32,33] a difference of at least 15% in degree of conversion between groups should be assessed to be clinically significant; so five posts for each luting cement were selected, and cemented with a different material (transparent shade) (Table 1): Calibra,[d] Multilink Automix,[b] and Variolink II.[b] Calibra is a high-resistance dual-light-curing/self-curing resin-based material. It consists mainly of dimethacrylate resins and bisphenol A-glycidyl methacrylate (Bis-GMA). Multilink Automix is a self-curing composite with a light-curing option. Its monomer matrix consists of dimethacrylate and 2-hydroxyethylmethacrylate (HEMA). Variolink II is a dual-curing composite whose organic matrix consists of Bis-GMA, urethane dimethacrylate, and 3-ethylene-glycol-dimethacrylate.

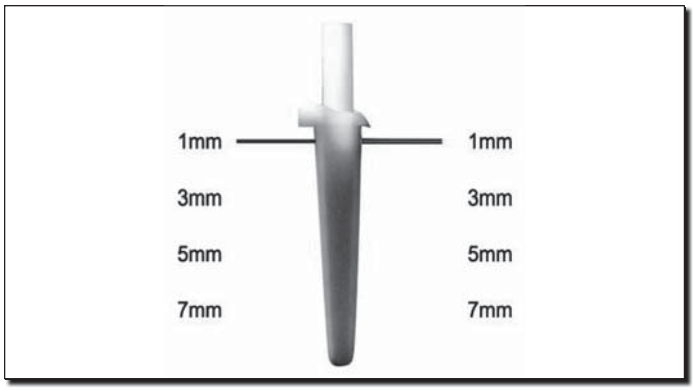

Fig. 1. Raman measurements were performed at increasing distances from the coronal-most portion of the glass-fiber post, in two positions on its surface.

Table 2. Mean DC, (in percentage), and standard deviation of the samples polymerized according to S (400 mW/cm^2 for 120 seconds) and H (1200 mW/cm^2 for 40 seconds) mode.

Curing modality		Calibra		Multilink Automix		Variolink II	
		S	H	S	H	S	H
Position	1 mm	61.5 (9)	68 (4.8)	75.8 (3.4)	67 (11.3)	76.9 (10.6)	68.7 (2.7)
	3 mm	59.9 (8.3)	67.9 (2.8)	72.7 (4.5)	66 (12.2)	65.4 (10.9)	67.8 (3)
	5 mm	60 (13.7)	68 (4.7)	68.7 (6.8)	59 (17.7)	62.4 (16.1)	65.1 (16)
	7 mm	56.9 (14.5)	68.7 (4.9)	67.4 (5.4)	56.8 (16.5)	60.3 (18.5)	55.1 (7.9)

Luting procedure - The post was layered with adhesive and the volatile component was removed by gentle air blowing. The self- and light-curing adhesive systems used were XP Bond+ SCA[d] for Calibra and Excite DSC[b] for Multilink Automix and Variolink II. The selected luting composite was mixed according to the manufacturers' instructions and extruded through a dedicated tip in order to completely fill the post space, then the post was positioned into the canal with care, to prevent void formation.

No adhesive system was placed into the canal walls to avoid the introduction of bias into the study.

Micro-Raman analysis - The samples were analyzed with a Dilor-HR-Labram Micro-Raman Spectrometer (laser, He:Ne; wavelength, 632.8 nm; numerical aperture 0.7) at x50 magnifications with an exposure time of 60 seconds.

The post surface was divided into zones A and B. For each zone, the specimens were examined at 1, 3, 5, and 7 mm, where 0 mm corresponds to the coronal-most part of the luting composite-covered post (Fig. 1). Eight spectra were collected for each sample at the previously mentioned positions, and then a baseline correction was applied. DC was determined as previously described.[32] The analysis did not inspect DC at 0 mm, since the DC in the coronal-most zone of the post could be affected by the presence of oxygen, which represents an uncontrollable variable.

Statistical analysis - The data were analyzed using ANOVA (P= 0.05) to test the null hypothesis, which stated that the result would not vary according to the treatments performed. After this, a post-hoc analysis was performed to determine which specific groups of measurements were significantly different (Student Neuman Keuls *t*-test, P= 0.05).

Results

Generally, the percentages of conversion seemed to be higher in the coronal-most aspects of the post, where the distance between composite and light tip was small, independent of the group examined (Table 2).

Examining each luting material (Fig. 2), Calibra had a constant behavior, not being particularly affected by the increase of the distance from the light tip. The DCs relative to the collections performed at 1, 3, 5, and 7 mm, both in Groups S and H, did not significantly differ from each other (P> 0.05).

Multilink Automix showed higher degrees of conversion in the most coronal portion of the post, with a significant difference among the samples belonging to Group S, with reference to the collections performed at 5 and 7 mm relative to those made at 1 mm.

On the contrary, Variolink II showed significant differences (P= 0.0002) in Group H among the percentages of conversion relative to farthest positions from the light source (7 mm).

Regarding the differences between the curing modalities (Groups H and S) with each material, the only significant difference between Groups S and H was found with Calibra at 3 mm (P= 0.04).

Discussion

This study compared how two different curing modalities affect the DC of different resin-based luting materials at 1, 3, 5, and 7 mm on the surface of a glass-fiber reinforced composite post, investigating on the effectiveness of high-power lamps. Those devices were developed in order to reduce the curing time, seen as a time-wasting procedure, but still some concerns remain on the quality of the polymer net produced by such a sudden activation and on the extent of the polymerization of the composite: the risk is to obtain polymers with scarcer physical properties than those cured for a longer time by means of standard power light-curing units.[35,36]

In regards to the evaluation of the behavior of dual-curing materials along the post surface, the present results showed that the distance from the light source affected the DC in almost all samples: the values at 7 mm were lower than at the coronal portion of the post, although the difference was weakly significant. For Calibra, the average difference in the percent conversion was 2.7%, whereas this value was greater in Multilink Automix and Variolink II (11.8 and 14.5%, respectively).

Calibra maintained a constant DC, showing homogeneous polymerization, independent of the test variables: this is reasonably caused by its peculiar composition. The other two composites showed a slightly lower percent conversion as increasing distances from the light tip, probably due to their photo-initiator content, although the conversion values were superior to 60%, independent of the curing modality used.

From a clinical perspective, we obtained the highest conversion rate using Calibra with H mode (1200 mW/cm$_2$ for 40 seconds), while when using Variolink II or Multilink Automix, S mode (400 mW/cm^2 for 120 seconds) is advisable.

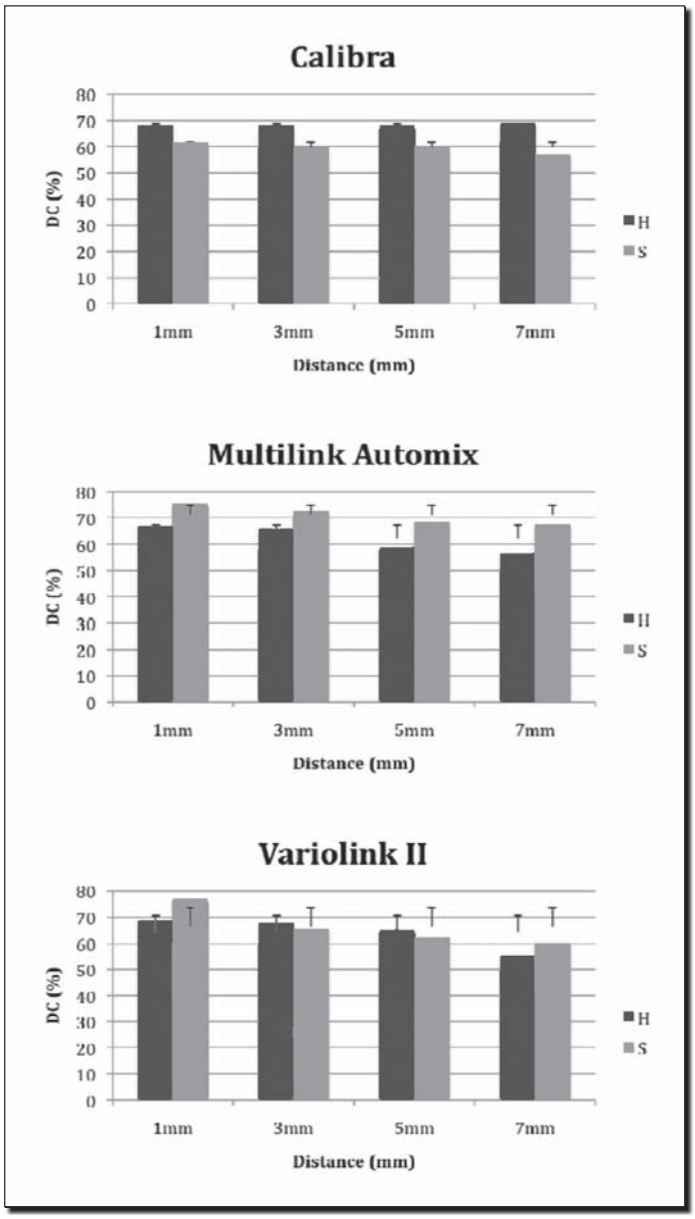

Fig. 2. Percentage degree of conversion reached by the tested materials when cured according to the H (1200 mW/cm^2 for 40 seconds) and the S mode (400 mW/cm^2 for 120 seconds). Increasing the distance from the light tip brings to a drawback in the material performances in all the tested composites except for Calibra.

The decrease in light intensity with increasing distance of the light tip from the composite surface, despite using a translucent light-conductive post occurs.[1] On this basis, dual-curing materials were tested to reduce the influence of this variable on DC.

A previous study[27] evaluated the DC in composites using light-transmitting posts, but it had limitations based on the specimens' size (longer and greater in diameter than a root canal), and the use of materials that cannot be used clinically. This study showed how a light-transmitting post increases the Knoop hardness number at greater depths near the post, but the results did not clearly demonstrate whether this device improved cure depth.

The present study was designed to reproduce the clinical conditions of cementation. The post protruded from the canal

so that dispersion of the radiant energy was a factor to be considered, and the light reached the composite by passing through the post. Therefore, our results are useful clinically.

No adhesive layer was applied onto the artificial canal walls; this choice is justified by the fact that the microRaman reads the surface DC of the material. Thus, since adhesion between adhesive and luting composite occurs, one could not discriminate whether the instrument evaluates the DC of the adhesive or the luting cement; then, adhesive would definitely have altered the results.

It is obvious that, in a clinical situation, this further element should be taken into account and could be an interesting element of future studies.

All the luting materials employed were in transparent shade: a spectrophotometric analysis might be necessary to determine the difference among the three transparent materials, but this was not in the aim of the study. The interest was mainly focused on the ability of the luting composite to convert adequately; from this point of view, the results obtained confirmed the variability in DC between the materials, although dual-curing materials are considered and the difference assessed were not relevant. Probably, these data cannot be extended to all the composites available on the market, since they are linked to their degree of transparency.

The decrease in DC at great distances from the light tip observed in dual-curing material, suggest that in glass-fiber post cementation a light-curing correct procedure should be followed for a reproducible conversion. Anyway, the distance from the light source clearly cannot be disregarded in post luting, independent of the nature of the composite used.

In this particular study we did not test a light-curing only and a self-curing material. This choice is justified by the fact that Vieno *et al*[33] clarified with the same type of analysis that light-curing composites, without any alternative activation, do not reach sufficient DCs when their distance from the light tip is greater than 2 mm for the power/time chosen in this study. In addition, it was not an aim of this study to compare self-curing materials, as these resin composites are difficult to handle during clinical procedures, demonstrated incompatibility with some adhesive systems and might have some drawbacks in the interfaces with fiber posts.[37,38]

The differences in DC between 1 and 7 mm highlights the need to improve the features of the post, which transmits some light along its axis only. Moreover, from a clinical point of view, creating deep post spaces, the clinician should be aware that the risk of insufficient luting composite features due to scarce monomer conversion could harm the long-term durability of the restorative procedure, since finite element analyses show that the stresses tend to concentrate in the apical-most areas.[34]

Considering the time and power of curing, the present results showed that these parameters must be optimized also for dual-cure composite materials.

a. Böhler Uddeholm Spa, Milan, Italy.
b. Ivoclar-Vivadent, Schaan, Liechtenstein.
c. EMS, Nyon, Switzerland.
d. Dentsply, Baillagues, Switzerland.

Acknowledgements: To the staff of the Laboratory of Chemistry for Technologies of the University of Brescia for their kind help in the use of the Micro-Raman.

Disclosure statement: The authors have no conflict of interest.

Dr. F. Cerutti is a PhD student, Department of Materials for Engineering, Dr. Depero is Professor and Head of the Department of Chemistry for Technologies, Faculty of Engineering, and Dr. A Cerutti is Professor and Director of the Department of Restorative Dentistry, Faculty of Medicine, University of Brescia, Italy. Dr. Gagliani is Professor, Department of Restorative Dentistry, Faculty of Medicine, University of Milan, Italy. Dr. Acquaviva is a PhD student, and Dr. Ferrari is Professor and Director of the Department of Dental Materials, University of Siena, Italy. Dr. Mangani is Professor, Department of Restorative Dentistry, Faculty of Medicine, University of Tor Vergata, Rome, Italy.

References

1. Assif D, Gorfil C. Biomechanical considerations in restoring endodontically treated teeth. *J Prosthet Dent* 1994;71:565-567.
2. Boschian Pest L, Cavalli G, Bertani P, Gagliani M. Adhesive post-endodontic restoration with fiber posts: Push-out tests and SEM observations. *Dent Mater* 2002;18:596-602.
3. Ferrari M, Cagidiaco MC, Grandini S, De Sanctis M, Goracci C. Post placement affects survival of endodontically treated premolars. *J Dent Res* 2007;86:729-734.
4. Malferrari S, Monaco C, Scotti R. Clinical evaluation of teeth restored with quartz reinforced-reinforced epoxy resin posts. *Int J Prosthodont* 2003;16:39-44.
5. Monticelli F, Grandini S, Goracci C, Ferrari M. Clinical behavior of translucent-fibre posts: A 2-year prospective study. *Int J Prosthodont* 2003;16:593-596.
6. Sorrentino R, Salameh Z, Zarone F, Tay F, Ferrari M. Effect of post-retained composite restoration of MOD preparations on the fracture resistance of endodontically treated teeth. *J Adhes Dent* 2007;9:49-56.
7. Lui JL. Depth of composite polymerization within simulated root canals using light-transmitting posts. *Oper Dent* 1994;19:165-168.
8. Mannocci F, Bertelli E, Sherriff M, Watson TF, Ford TR. Three-year clinical comparison of survival of endodontically treated teeth restored with either full cast coverage or with direct composite restoration. *J Prosthet Dent* 2002;88:297-301.
9. Tay FR, Loushine RJ, Lambrechts P, Weller RN, Pashley DH. Geometric factors affecting dentin bonding in root canals: A theoretical modeling approach. *J Endod* 2005;31:584-589.
10. Debnath S, Ranade R, Wunder SL, McCool J, Boberick K, Baran G. Interface effects on mechanical properties of particle-reinforced composites. *Dent Mat*er 2004;20;677-686.
11. Roulet JF. *Degradation of posterior composite resins in vivo. Degradation of dental polymers*. Basel: Karger, 1987; 161-214.
12. Bhamra Gurcharn S, Fleming GJP. Effects of halogen light irradiation variables (tip diameter, irradiance, irradiation protocol) on flexural strength properties of resin-based composites. *J Dent* 2008;36:643 - 650.
13. Lohbauer U, Rahiotis C, Krämer N, Petschelt A, Eliades G. The effect of different light-curing units on fatigue behavior and degree of conversion of a resin composite. *Dent Mater* 2005;21:608-615.
14. Halvorson RH, Erickson RL, Davidson CL. Energy dependent polymerization of resin-based composite. *Dent Mater* 2002;18:463-469.
15. Peutzfeldt A, Asmussen E. Resin composite properties and energy density of light cure. *J Dent Res* 2005;84;659-662.
16. Aravamudhan K, Rakowski D, Fan PL. Variation of depth cure and intensity with distance using LED curing lights. *Dent Mater* 2006;22:988-994.
17. Felix CA, Price RBT. The effect of distance from light source on light intensity from curing lights. *J Adhes Dent* 2003;4:283-291.
18. Yap AJ. Effectiveness of polymerization in composite restoratives claiming bulk placement: Impact of cavity depth and exposure time. *Oper Dent* 2000;25:113-120.
19. Ferrari M, Vichi A, Mannocci F, Mason PM. Retrospective study of the clinical performance of fiber posts. *Am J Dent* 2000;13:9B-13B.
20. Roberts HW, Leonard DL, Vandewalle KS, Cohen ME, Charlton DG. The effect of a translucent post on resin composite depth of cure. *Dent Mater* 2004;20:617-622.
21. Strassler HE, Coletti P, Hutter J. Composite polymerization within simulated root canals using light-transmitting posts. *J Dent Res* 1997;76:(Abstr 551).
22. Yoldas O, Alaçam T. Microhardness of composites in simulated root canals cured with light transmitting posts and glass-fiber reinforced composite posts. *J Endod* 2005;31:2,104-105.
23. Lindberg A, Peutzfeldt A, van Dijken JVW. Effect of power density of curing unit exposure duration, and light guide distance on composite depth of cure. *Clin Oral Invest* 2005;9:71-76.
24. Braga RR, César PF, Gonzaga CC. Mechanical properties of resin cements

with different activation modes. *J Oral Rehabil* 2002;29:257-266.
25. Kumbuloglu O, Lassila LV, Vallittu PK. A study of the physical and chemical properties of four resin composites luting cements. *Int J Prosthodont* 2004;17:357-363.
26. Chen YC, Ferracane JL, Prahl AS. A pilot study of a simple photon migration model for predicting depth of cure in dental composite. *Dent Mater* 2005;21:1075-1086.
27. Miyazaki M, Onose H, Iida N, Kazama H. Determination of residual double bonds in resin-dentin interface by Raman spectroscopy. *Dent Mater* 2003;19:245-251.
28. Faria e Silva AL, Gallego Arias V, Silva Soares LE, Abrahao Martin A, Marcondes Martins LR. Influence of post-post translucency on the degree of conversion of a dual-cured resin cement. *J Endod* 2007;33:303-305.
29. Väkiparta M, Puska M, Vallittu PK. Residual monomers and degree of conversion of partially bioresorbable fiber-reinforced composite. *Acta Biomater* 2006;2:29-37.
30. Beun S, Glorieux T, Devaux J, Vreven J, Leloup G. Characterization of nanofilled compared to universal and microfilled composites. *Dent Mater* 2007;23:51-59.
31. Atai M, Nekoomanesh M, Hashemi SA, Amani S. Physical and mechanical properties of an experimental dental composite based on a new monomer. *Dent Mater* 2004;20:663-668.
32. Acquaviva PA, Cerutti F, Adami G, Gagliani M, Ferrari M, Gherlone E, Cerutti A. Degree of conversion of three composite materials employed in the adhesive cementation of indirect restorations: A micro-Raman analysis *J Dent* 2009;37:610-615.
33. Vieno S, Madini L, Barabanti N, Alessandri I, Gagliani M, Cerutti A. Indirect resin composite restorations: Evaluation of polymerization of luting agents by means of micro-Raman spectrophotometry. *Minerva Stom* 2009;58:1-8. (In Italian).
34. Boschian Pest L, Guidotti S, Pietrabissa R, Gagliani M. Stress distribution in a post-restored tooth using the three-dimensional finite element method. *J Oral Rehabil* 2006;33:690-697.
35. Braga R, Ballester RY, Ferracane J. Factors involved in the development of polymerization shrinkage stress in resin-composites: A systematic review. *Dent Mater* 2005;21:962-970.
36. Calheiros FC, Daronch M, Rueggeberg FA, Braga RR. Influence of irradiant energy on degree of conversion, polymerization rate and shrinkage stress in an experimental resin composite system. *Dent Mater* 2008;24:1164-1168.
37. Arrais CAG, Rueggeberg FA, Waller JL, de Goes MF, Giannini M. Effect of curing mode on the polymerization characteristics of dual-cured resin cement systems. *J Dent* 2008;36:418-426.
38. Ceballos L, Garrido MA, Fuentes V, Rodriguez J. Mechanical characterization of resin cements used for luting fiber posts by nanoindentation. *Dent Mater* 2007;23-100-105.