

# Influence of viscosity and curing mode on degree of conversion of dual-cured resin cements

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## ABSTRACT

**Objective:** The purpose of this study was to evaluate the effects of the viscosities and curing modes on the degree of conversion (DC) of two resin cements.

**Methods:** Eight experimental groups were evaluated (n=5), according to the dual-cured resin cements (Nexus 2/Variolink II), viscosity (low and high) and evaluation time (5 minutes and 24 hours). The resin cements were applied to surface of a horizontal attenuated-total-reflectance unit and were polymerized either with self-cure (SC) or light exposure (XL3000/3M ESPE) for 40 seconds. Infrared spectra were obtained after 5 minutes and 24 hours (Nicolet 520 FT-IR/Thermo Scientific Inc.). DC was calculated according to changes in aliphatic-to-aromatic peak ratios pre- and post-curing. Data (%) were analyzed by 3-way repeated measure ANOVA (curing mode, viscosity and time interval) and Tukey's post-hoc test (P<.05).

**Results:** The dual-polymerizing mode provided higher DC than auto-polymerization. The DC mean values increased for both resin cements after 24 hours. The low-viscosity resin cements from light-activated or self-cured groups exhibited higher DC than high viscosity version.

**Conclusion:** The DC of resin cements was higher for the low viscosity version, following the light-polymerization and when were tested after 24 hours. (Eur J Dent 2013;7:81-85)

**Key words:** Resin cements; curing mode; degree of conversion

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## INTRODUCTION

Dual-polymerizing resin cements have been extensively used for placement of indirect restorations and posts. The dual-polymerizing materials were developed to compensate for the lack of polymerization in the absence of light and to represent a combination of auto- and light-polymerizing components. In some clinical situations, such as in dark zones at the apical region and during the cementation of indirect restorations, the severe light attenuation results in low degree of conversion (DC), which can compromise the mechanical properties and consequently the longevity of the indirect restorations.<sup>1-4</sup>

Modifications in the viscosity of the resin cements allow their use in different clinical situations. The option for low viscosity versions offer some benefits, such as minor thickness of the pellicle that was formed following the restoration placement. The lowest film thickness generates smaller polymerization shrinkage, reducing the possibility of gaps formation and premature marginal leakage.<sup>5,6</sup> The difference in the cement formulations that change the viscosity is related to the proportion between resin matrix and filler particle content.<sup>7</sup>

High volume fraction of fillers can increase the viscosity and the elastic modulus and strength of composites.<sup>7,8</sup> Low viscosity or flowable resins and resin cements present lower filler loading than regular restorative materials. Most direct dental restorative composites use bisphenol-A-diglycidylether dimethacrylate (Bis-GMA), which is considered a very viscous monomer, and when mixed with higher filler loadings, it becomes a nearly solid mass and unusable product. Vinyl groups (e.g., ethylene glycol dimethacrylate) are added as a thinner or diluent monomer for uncured pastes, which are considered another approach to change the viscosity of resin-based materials. The filler loading and the viscosity of composites may interfere in the monomer conversion, since they could restrict the mobility of monomers and the propagation of polymerization reaction.<sup>7,9-11</sup>

The restorative resin-based materials must reach a high degree of monomer conversion in order to present better clinical performance and longevity and also to reduce the early failures. For the dual-polymerizing resin cements, the self-cure mode should ensure the high level of conver-

sion, especially in the cervical proximal areas, the root canal and in the internal and deep areas of the cavity preparations.<sup>12-14</sup> Several methodologies have been used to analyze the DC of resin-based materials; however, most of them use Fourier Transform Infrared Spectroscopy (FTIR).

Although many researchers have evaluated the polymerization effectiveness to determine the physical and mechanical properties of dental materials,<sup>9,15,16</sup> little information is known about the influence of different viscosities of dual-cured resin cements on their physical properties, such as the degree of conversion in situations of self- and dual-polymerization. Thus, the purpose of this study was to measure the DC of two commercially dual-cured resin cements in different viscosities (low and high) when they were light-activated or when the materials were allowed to self-cure solely, after 5 minutes and 24 hours from the mixture of pastes (base and catalyst). The hypothesis tested was that curing mode, evaluation time and viscosity would affect the DC of the resin cements.

## METHODS & MATERIALS

Two commercial dual-cure resin cements in high and low viscosities were evaluated: Variolink II (Ivoclar Vivadent, Schaan, Liechtenstein) and Nexus 2 (Kerr Corp., Orange, CA, USA). The compositions of the two resin cements tested are presented in Table 1. The resin cements consist of two paste components that were equally dispensed and mixed together according to manufacturers' instructions.

After mixing, resin cements (n=5) were applied to the horizontal attenuated total reflectance ZnSe crystal at 45° (Fourier transform infrared spectrometer, FT-IR Spectrometer 520, Nicolet Instrument Corp, Madison, WI, USA) at room temperature. Adhesive tape (3M, Sumaré, SP, Brazil) was placed on the ZnSe crystal surface to act as a spacer, ensuring standard thickness for all specimens (100-120 mm). A Mylar strip (Quimidrol Com. Ind. Imp. Ltda, Joinville, SC, Brazil) was placed over the cement and was pressed flat to spread the material on the crystal surface. The spectrum of unpolymerized material was obtained and the cements were either light-cured (dual-polymerizing mode) or allowed to self-cure only (autopolymerizing mode). Each specimen was left on the crystal surface and further spectra were

obtained 5 minutes and 24 hours after post mixing. For light-cured groups, a halogen light curing unit (XL 3000, 3M ESPE, St. Paul, MN, USA) was used during 40 seconds ( $\pm 600 \text{ mW/cm}^2$ ). The light intensity was periodically checked with a radiometer (Curing Radiometer, model 100, Kerr Corp., Orange, CA, USA).

Spectra were obtained between  $4000 \text{ cm}^{-1}$  and  $750 \text{ cm}^{-1}$  at a resolution of  $4 \text{ cm}^{-1}$ . Monomer conversion was calculated (%) according to the changes in the ratio between the absorbance peaks corresponding to the aliphatic (C=C) ( $1638 \text{ cm}^{-1}$ ) and aromatic ( $1608 \text{ cm}^{-1}$ ) carbon double bonds prior to and 5 minutes and 24 hours after polymerization initiation for both curing modes.<sup>10</sup> The aromatic peak is used as an internal reference because the intensity does not change during the polymerization reaction.<sup>9,11</sup>

The effect of curing mode, viscosity and time intervals on the DC were evaluated for each material. Three-way repeated measure analysis of variance (ANOVA) (curing mode, viscosities and evaluation time) was performed and Tukey's post-hoc test was used to detect pair wise differences among experimental groups. The data was analyzed using the statistical program SAS 9.1 (SAS Institute, Cary, NC, USA) and all statistical testing was performed at a pre-set alpha of 0.05 ( $P < .05$ ).

## RESULTS

The DC means for each product are described in Tables 2 and 3. The statistical analysis of the data showed significant differences in curing mode, viscosity and evaluation time factors for both resin cements ( $P = .01$ ). There was significant interaction between factors: the curing mode x

Table 1. Compositions of the resin cements used in this study.

Resin cement (Manufacturer)	Composition	Lote number (L: low; H: high)
Nexus 2	Base: Bis-GMA, camphoroquinone, barium aluminoborosilicate glass. Catalyst: Bis-GMA, TEGDMA, barium aluminoborosilicate glass (70% by weight and 53% by volume of fillers particles).	438681 (base) 452344 (L) and 452365 (H)
Variolink II	Base: Bis-GMA (10-25%), urethane dimethacrylate (2.5-10%), TEGDMA (2.5-10%), inorganic filler, ytterbium trifluoride, initiator, stabilizer. Catalyst: Bis-GMA (50-100%), urethane dimethacrylate (2.5-10%), TEGDMA (2.5-10%), inorganic filler, ytterbium trifluoride, benzoyl peroxide (<2.5%), stabilizer. Low-viscosity: 27.9% by weight of monomers and 71.2% by weight or 43.6% by volume of fillers particles. High viscosity: 22% by weight of monomers and 77% by weight or 52% by volume of fillers particles.	J24363 (base) J19103 (L) and J19730 (H)

Abbreviations: Bis-GMA: bisphenol-A-diglycidylether dimethacrylate; TEGDMA: triethylene glycol dimethacrylate.

Table 2. Degree of conversion (%) means (SD) for Nexus 2 resin cement.

Viscosity	Time	Activation mode	
		Dual-polymerized	Auto-polymerized
High *	5 min	63.5 (1.9) Ba	44.7 (2.1) Bb
	24 h	86.8 (1.5) Aa	77.3 (1.1) Ab
Low ●	5 min	64.4 (1.8) Ba	48.6 (3.2) Bb
	24 h	89.9 (0.9) Aa	78.8 (0.8) Ab

Groups having similar letters are not significantly different, upper case (column): compare times within viscosity and curing mode and lower case (row): compare curing modes within time and viscosity. Viscosities having different symbols are significantly different.

Table 3. Degree of conversion (%) means (SD) of Variolink II resin cement.

Viscosity	Time	Activation mode	
		Dual-polymerized	Auto-polymerized
High *	5min	62.3 (1.8) Ba	43,7 (2.0) Bb
	24h	84.9 (1.4) Aa	75.8 (1.3) Ab
Low ●	5min	63.7 (1.7) Ba	46.1 (2.0) Bb
	24h	87.7 (1.2) Aa	77.5 (1.3) Ab

Groups having similar letters are not significantly different, upper case (column): compare times within viscosity and curing mode and lower case (row): compare curing modes within time and viscosity. Viscosities having different symbols are significantly different.

evaluation time ( $P=.01$ ). Other double-interactions and the triple-interaction (curing mode x viscosity x evaluation time) were not significant ( $P>.05$ ).

The DC of resin cements was higher when the materials were light-cured and when the low-viscosity versions were used than when self-curing mode and the high-viscosity resin cements were tested, respectively ( $P<.05$ ). The elapsed 24 hours after curing increased the DC ( $P<.05$ ).

## DISCUSSION

The results of this study showed that the curing mode (auto- and dual-polymerization), the viscosity (low or high) and the evaluation time (5 minutes or 24 hours) influenced the DC of resin cements. Thus, the research hypothesis stating that these factors would result in significant changes in the DC was accepted. As the DC is dependent on the monomeric composition and the concentration and type of the photoinitiator in each product,<sup>9,17</sup> the statistical analysis comparing the DC between resin cements must not be made (i.e., the materials were analyzed separately).

The main factors under study (curing mode, viscosities and time intervals) produced similar effects for the resin cements, which presented different formulations regarding the resin matrix composition and type and concentration of filler particles. The variations on the proportion of components of resin matrix and filler particles that produced the low and the high versions caused no difference in behavior between the two cements results.<sup>7</sup>

A high monomer conversion with cross-linked polymeric network formation is essential for the durability of the resin-based restorations.<sup>9-11</sup> Studies have shown the importance of a high DC of resinous materials in order to reach better mechanical properties, since the presence of a high amount of residual methacrylate monomers compromises the hardness, the abrasion and the fracture resistance. Moreover, the low DC can increase the sorption and the solubility, interfering in the color stability and mass loss of the resin cement.<sup>2,4,9,14,17-20</sup>

The resin cements showed significant difference between the evaluation times, 5 minutes and 24 hours from the polymerization initiation, independently of viscosity or curing mode. Thus, the polymerization reaction for both resin cements con-

tinued after 5 minutes of the mixing of the base and catalyst pastes. The continuation of the chemical part of the polymerization reaction (benzoyl peroxide + tertiary amines) was primarily responsible for the increase of the DC after 24 hours. Additionally, the percentage of DC increasing after 24 hours was higher for the self-cure mode than the dual cure mode, which ranged from 38.3 to 42.4% and from 26.6 to 28.4%, respectively. The continuation reaction 24 hours after polymerization initiation resulted in a reduction of absorbance peaks that corresponded to the aliphatic carbon double bonds<sup>10</sup> and was detected in this study.

The dual polymerization increased DC, which corroborates with several studies that compared the polymerization modes for resin-based materials.<sup>2,11,21,22</sup> For both resin cements used in this study, the direct light-polymerization provided DC that was higher and ranged from 10 to 30% higher than those observed for the self-curing mode. Also, it was higher at 5 minutes (24 to 30%) than for 24 hours (10 to 12%) from the polymerization initiation. The auto-polymerization may be insufficient to result in an adequate DC for the long lasting restorations. Clinically, the low viscosity version ensured higher DC, and 24 hours elapsed time after setting of restoration is appropriate for the final appointment, since the higher DC could better support the final occlusal adjustments, finishing, and polishing procedures. The direct light-polymerization can be considered a limitation of this study, since the light is applied directly to the resin cement layer only at the periphery of restorations. On the other hand, the inner part of indirect restorations is reached by an attenuated light and resin cement may depend only on self-cure activation.

Regarding the differences in the DC between high and low viscosities, higher mean values were found for low viscosity versions of both resin cements. Low viscosity materials can be due to lower filler loading and/or higher addition low viscosity diluents monomers. The low viscosity monomer allows better mobility and distribution of free radicals inside the resin material, which can increase the polymerization reaction and the monomer conversion.<sup>9</sup> The increase of DC promoted by the low viscosity versions in percentage, following the polymerization mode ranged from 0.9 to 3.1% for the

dual-polymerizing groups and ranged from 1.5 to 3.9% for the auto-polymerizing mode, which was the highest percentage among the groups from Nexus 2 resin cement. For the Variolink II, dual-polymerizing groups ranged from 1.4 to 2.8%, while the auto-polymerizing mode ranged from 1.7 to 2.4%.

## CONCLUSION

The direct light-activation and the low viscosity version were important to provide higher DC for the dual-polymerizing resin cements. Twenty-four hours after seating the indirect restoration is appropriate for final occlusal adjustments, finishing, and polishing procedures, which are capable of generating stress to the resin cement layer.

## REFERENCES

1. Sigemori RM, Reis AF, Giannini M, Paulillo LA. Curing depth of a resin-modified glass ionomer and two resin-based luting agents. *Oper Dent* 2005;30:185-189.
2. Arrais CA, 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* 2005;36:418-426.
3. Arrais CA, Giannini M, Rueggeberg FA. Effect of sodium sulfinate salts on the polymerization characteristics of dual-cured resin cement systems exposed to attenuated light-activation. *J Dent* 2009;37:219-227.
4. Arrais CA, Giannini M, Rueggeberg FA. Kinetic analysis of monomer conversion in auto- and dual- polymerizing modes of commercial resin luting cements. *J Prosthet Dent* 2009;101:128-136.
5. Oliveira LC, Duarte S Jr, Araujo CA, Abrahão A. Effect of low-elastic modulus liner and base as stress-absorbing layer in composite resin restorations. *Dent Mater* 2010;26:159-169.
6. Lutz F, Krejci I, Barbakow F. Quality and durability of marginal adaptation in bonded composite restorations. *Dent Mater* 1991;7:107-113.
7. Darvell BW. Resin Restorative Materials. In: Darvell BW, eds. *Materials Science for Dentistry*. Hong Kong: B W Darvell. 2000:111-139.
8. Kim KH, Ong JL, Okuno O. The effect of filler loading and morphology on the mechanical properties of contemporary composites. *J Prosthet Dent* 2002;87:642-649.
9. Ferracane JL, Greener EH. Fourier transform infrared analysis of degree of polymerization in unfilled resins-methods comparison. *J Dent Res* 1984;3:1093-1095.
10. Ruyter IE, Svendsen AS. Remaining methacrylate groups in composite restorative materials. *Acta Odontol Scand* 1978;36:75-82.
11. Rueggeberg FA, Caughman WF. The influence of light exposure on polymerization of dual-cure resin cements. *Oper Dent* 1993;18:48-55.
12. Blackman R, Barghi N, Duke E. Influence of ceramic thickness on the polymerization of light-cured resin cement. *J Prosthet Dent* 1990;63:295-300.
13. El-Mowafy OM, Rubo MH, El-Badrawy WA. Hardening of new resin cements cured through a ceramic inlay. *Oper Dent* 1999;24:338-343.
14. Hofmann N, Papsthart G, Hugo B, Klaiber B. Comparison of photo-activation versus chemical or dual-curing of resin-based luting cements regarding flexural strength, modulus and surface hardness. *J Oral Rehabil* 2001;28:1022-1028.
15. Arrais CA, Giannini M, Rueggeberg FA, Pashley DH. Microtensile bond strength of dual polymerizing cementing systems to dentin using different polymerizing modes. *J Prosthet Dent* 2007;97:99-106.
16. Menezes MJ, Arrais CA, Giannini M. Influence of light-activated and auto- and dual-polymerizing adhesive systems on bond strength of indirect composite resin to dentin. *J Prosthet Dent* 2006;96:115-121.
17. Asmussen E. Restorative resins: hardness and strength vs. quantity of remaining double bonds. *Scand J Dent Res* 1982;90: 484-489.
18. El-Mowafy OM, Rubo MH. Influence of composite inlay/onlay thickness on hardening of dual-cured resin cements. *J Can Dent Assoc* 2000;66:147-148.
19. Blackman R, Barghi N, Duke E. Influence of ceramic thickness on the polymerization of light-cured resin cement. *J Prosthet Dent* 1990;63:295-300.
20. Hasegawa EA, Boyer DB, Chan DC. Hardening of dual-cured cements under composite resin inlays. *J Prosthet Dent* 1991;66:187-192.
21. Darr AH, Jacobsen PH. Conversion of dual cure luting cements. *J Oral Rehabil* 1995;22:43-47.
22. Caughman WF, Chan DC, Rueggeberg FA. Curing potential of dual-polymerizable resin cements in simulated clinical situations. *J Prosthet Dent* 2001;86: 101-106.