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ORIGINAL ARTICLE

The effect of curing light and chemical catalyst on the degree of conversion of two dual cured resin luting cements

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Abstract The aim of this study was to evaluate the influence of different curing lights and chemical catalysts on the degree of conversion of resin luting cements. A total of 60 disk-shaped specimens of RelyX ARC or Panavia F of diameter 5 mm and thickness 0.5 mm were prepared and the respective chemical catalyst (Scotchbond Multi-Purpose Plus or ED Primer) was added. The specimens were light-cured using different curing units (an argon ion laser, an LED or a quartz-tungsten-halogen light) through shade A2 composite disks of diameter 10 mm and thickness 2 mm. After 24 h of dry storage at 37°C, the degree of conversion of the resin luting cements was measured by Fourier-transformed infrared spectroscopy. For statistical analysis, ANOVA and the Tukey test were used, with $p \leq 0.05$. Panavia F when used without catalyst and cured using the LED or the argon ion laser showed degree of conversion values significantly lower than RelyX ARC, with and without catalyst, and cured with any of the light sources. Therefore, the degree of conversion of Panavia F with ED Primer cured with the quartz-tungsten-halogen light was significantly different from that of RelyX ARC regardless of the use of the chemical catalyst and light curing source. In conclusion, RelyX ARC can be cured satisfactorily with the argon ion laser, LED or quartz-tungsten-halogen light with or without a chemical catalyst. To obtain a satisfactory degree of conversion, Panavia F luting cement should be used with ED Primer and cured with halogen light.

Keywords Curing light · Degree of cure · Resin cement · Photoactivation

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Introduction

With the new developments in adhesive dentistry, resin luting cements have become widely used due to their ability to bond indirect restorations to the tooth structure [1, 2]. These materials can minimize some adverse effects of direct composite restorations, such as polymerization contraction stress [3] and gap formation at the tooth/restoration interface [4]. Therefore, the procedure involving resinous luting of thick and opaque indirect restorations and fiber posts needs an adequate cure to reach a satisfactory degree of conversion of the cement used [2, 5]. The degree of conversion is affected by the attenuation of the light that reaches the resin cement, by the distance of the light source tip, irradiance and energy density applied, and by the transmission properties of the indirect restoration [5]. An insufficient degree of conversion can lead to deficient mechanical properties of the resin luting cement, including inadequate hardness [6, 7], wear resistance [8] and water sorption, residual monomer [9], and problems with biocompatibility. A low degree of resin polymerization increases the risk of restoration failure and marginal fracture, gap formation and the occurrence of secondary caries [4], and reduces the retention of the indirect restoration or the post [10].

In an attempt to overcome these problems, dual curing resin cements were developed to combine important properties of chemical-cured and light-cured materials so as to provide efficient cure in deeper areas and places with reduced light penetration [2, 11]. However, in some situations, the polymerization reaction may be mainly activated by a chemical mechanism (self-cure). Chemical catalysts have been used with luting materials with the aim of promoting better resin cement properties [2]. These catalyst formulations contain coinitiator systems, such as tertiary amine/benzoyl peroxide, that may improve the rate of monomer conversion when mixed with the material, especially in regions with poor light penetration [2].

On the other hand, choosing the correct light source for curing is important to achieve a satisfactory degree of cure of the resin luting cement. Traditionally, quartz-tungsten-halogen (QTH) lights have been widely used in dental practice. They have a spectral wavelength in the range 400–500 nm and an irradiance in the range 300–1,000 mW/cm^2 [12, 13]. However, they have some drawbacks, such as gradual reduction in irradiance over time, the need for filters for wavelength selection, limited depth of cure, and considerable heat generation [14, 15]. Blue light emitting diode (LED) units have a narrow spectral range, which targets the absorption wavelength of camphorquinone, with a peak value of 468 nm, allowing low amounts of wasted energy and minimum heat generation [14, 16]. It has been claimed that this could provide higher monomer conversion efficiency and reduce the exposure time required [17]. Studies have shown that current LED curing units can replace, in most situations, conventional QTH light units [15, 18].

The argon ion laser has arisen as an alternative light source for polymerizing resin composites, resin luting cements, and for bleaching procedures [19–21]. For camphorquinone-based resins, the activation emits a blue light in the wavelength range 400–500 nm with peak values at about 468 and 480 nm, depending on the monomer formulation, and it has an irradiance at up to 2,000 mW/cm^2 [21–24]. Furthermore, the argon ion laser has certain characteristics, such as low beam divergence, collimation, monochromaticity, coherency, absorption selectivity and fiber delivery ability, that make it particularly suitable for clinical use to cure resinous materials with a greater degree of cure and improved physical and mechanical properties [22, 23, 25]. Although these laser units may accelerate the cure of composites, they can promote a temperature increase at the light-curing tip [21].

The effect of the chemical catalyst on the properties of resin luting cements has not yet been clarified. Also, determining the most effective light source for curing resin cements still needs more study. Therefore, the aim of this study was to evaluate the influence of the incorporation of a chemical catalyst on cement manipulation and different light curing units on the degree of conversion of resin luting cements.

Materials and methods

Experimental design

The dual cured resin luting cement Panavia F (Kuraray Medical, Tokyo, Japan) and RelyX ARC (3 M ESPE, St. Paul, MN), with their respective chemical catalysts incorporated by manipulation of the material, were tested. The composition of the materials is shown in Table 1. A total of 60 disk-shaped specimens of diameter 5 mm and thickness 0.5 mm were prepared from the resin cements using the respective chemical catalysts. For specimen preparation, the cements were placed in a silicon mold (Express XT; 3 M ESPE, St Paul, MN) and then covered with Mylar film and a glass slide, in order to form a flat surface.

For Panavia F alone, equal amounts (27 g) of base and catalyst were weighed on an analytical balance [2], mixed for 15 s, and then immediately photocured. In the groups to which the ED Primer had been added, one drop of each of the primer liquids A and B was mixed for 10 s, and gently air-dried for 5 s for solvent evaporation. Thereafter, 1.4 mg of this solution was mixed with equal amounts (27 g) of base and catalyst of Panavia F for 15 s, and immediately photocured [2].

Specimens of RelyX ARC alone were prepared in the same way as the Panavia F specimens, with equal amounts (27 g) of base and catalyst, mixed for 15 s and then photocured. For specimens with the added chemical catalyst (catalyst of Scotchbond Multi-Purpose), 1.4 mg of the liquid was mixed with equal amounts (27 g) of base and catalyst of RelyX ARC for 15 s, and immediately photocured.

A disk (10 mm in diameter and 2 mm thick) of a nanofilled resin composite shade A2 (Filtek Z350; 3 M ESPE) was prepared to simulate an indirect restoration. The resin luting cements were light-cured through the composite for 40 s, using three light sources: argon ion laser at 600 mW/cm^2 (Accucure 3000/Lasermed); LED at 1,400 mW/cm^2 (FLASHlite 1401/Discus Dental); and QTH light at 600 mW/cm^2 (VIP/Bisco, Schaumburg, IL). The irradiance of all curing sources was measured with a radiometer before light activation (Demetron Kerr Corporation, Orange, CA). The light spectra of all curing units are shown in Fig. 1.

Degree of conversion

After 24 hours of dry storage at 37°C in the dark, the degree of conversion (DC) of the specimens was deter-

Table 1 Materials used in the study with composition and manufacturer's information

Resin cements	Composition	Manufacturer
Panavia F	Paste A (batch 00250B): 10-Methacryloyloxydecyl dihydrogen phosphate, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate, silanated sílica, photoinitiators, DL-camphorquinone, benzoyl peroxide Paste B (batch 00027B): Hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic dimethacrylate. Sodium aromatic sulfinate, accelerator, sodium fluoride, silanated barium glass	Kuraray Medical, Tokyo, Japan
ED Primer	Primer A (batch 00272A): 2-Hydroxyethyl methacrylate, methacryloyloxydecyl dihydrogen phosphate, NM-aminosalicylic acid, diethanol-*p*-toluidine, water Primer B (batch 00147A): NM-Aminosalicylic acid, T-isopropylic benzenic sodium sulfinate, diethanol-*p*-toluidine, water	Kuraray Medical, Tokyo, Japan
RelyX ARC (batch GU9JG)	Paste A: Silane-treated ceramic, bis-glycidyl methacrylate, triethylene glycol dimethacrylate, photoinitiators, amine, silane-treated silica, functionalized dimethacrylate polymer Paste B: Silane-treated ceramic, triethylene glycol dimethacrylate, bis-glycidyl methacrylate, silane-treated silica, benzoyl peroxide, functionalized dimethacrylate polymer	3 M ESPE, St Paul, MN
Catalyst for Scotchbond Multi-Purpose (batch 9BE)	Bisphenol A diglycidyl ether dimethacrylate, 2-hydroxyethyl methacrylate, benzoyl peroxide	3 M ESPE, St Paul, MN

mined using Fourier transform infrared spectroscopy (FTIR) with a Spectrum 100 instrument (PerkinElmer, Shelton, CT). For each specimen 32 spectra were analyzed. The ratio between the intensities of aliphatic C=C (at 1,637.3 cm^{-1}) and aromatic C=C (at 1,608.3 cm^{-1}) peaks for uncured and cured samples were used to calculate the degree of conversion, according to the following equation:

$$DC = \left[1 - \left(\frac{[\text{Abs (C = C aliph)/Abs (C}\cdots\text{C arom)] cured resin}}{[\text{Abs (C = C aliph)/Abs(C}\cdots\text{C arom)] uncured risen}}\right)\right] \times 100$$

where DC is the degree of conversion, Abs(C=C arom) is the height of the benzene ring peak, and Abs(C=C aliph) is the height of the aliphatic C=C bond peak, for both cured and uncured composites.

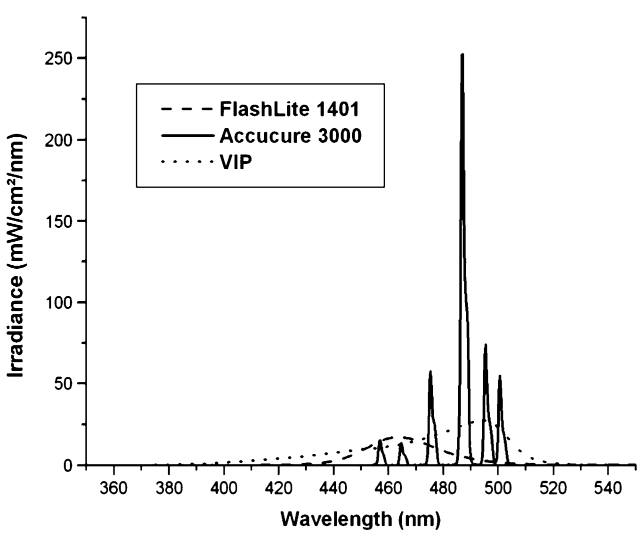

Fig. 1 Spectra of the light curing units used

A one-way ANOVA was performed to compare the differences among the tested groups at a preset alpha of 0.05, followed by Tukey's post hoc test.

Results

Means and standard deviations of the degree of conversion of the resin luting cements are shown in Table 2. Panavia F photocured with the LED and with the argon ion laser without chemical catalyst exhibited the lowest degree of conversion, which was significantly different from that of the other groups tested ($p \leq 0.05$). For the RelyX luting cement, no statistically significant differences in the degree of conversion were found between the light sources and the presence of chemical catalyst on manipulation. Photopolymerization with the QTH light showed better degree of conversion, which was significantly different from the degree of conversion with the LED and with the laser for Panavia F without catalyst incorporation. However, for RelyX ARC alone, there were no differences between the degrees of conversion achieved with any of the light curing units ($p \geq 0.05$). Figures 2 and 3 show the means of the degrees of conversion for Panavia F and RelyX ARC.

Discussion

The resin luting cements used in this study (RelyX ARC and Panavia F) are widely used in clinical practice for cementing fiber posts and indirect restorations. Most resin luting materials contain a diketone photoinitiator, such as camphorquinone with a broad absorption spectral peak at

Table 2 Degrees of (percentage) conversion of the tested groups. Values are means (standard deviations)

Group	Degree of conversion (%)
1. RelyX ARC+LED	68.68 (9.10) A
2. RelyX ARC+Laser+catalyst	67.58 (5.84) A
3. RelyX ARC+LED+catalyst	62.0 (4.73) AB
4. RelyX ARC+Laser	60.4 (10.1) AB
5. RelyX ARC+QTH	59.2 (11.3) AB
6. RelyX ARC+QTH+catalyst	54.26 (5.84) ABC
7. Panavia F+QTH+catalyst	54.1 (5.61) ABC
8. Panavia F+QTH	44.9 (9.49) BC
9. Panavia F+LED+catalyst	42.0 (6.23) BC
10. Panavia F+Laser+catalyst	35.6 (3.07) C
11. Panavia F+LED	10.91(8.68) D
12. Panavia F+Laser	7.10 (5.53) D

Different letters mean statistical significant difference among the groups

468 nm in the blue region of the visible spectrum [26, 27]. However, the spectral distribution of the output from the curing light source and the maximum absorption peak of a photoinitiator can affect the chemical and physical properties of a given resin.

Some resinous photocure or dual cure materials, such as resin cements, may have a lower concentration of camphorquinone or other photoinitiator such as phenyl propanedione [28] (PPD) and monoacrylphosphine oxide [29] (TPO), and sometimes the percentage of photoinitiator is not clarified by the manufacturer because a patent on the product has been applied for. For Panavia F, the manufacturer omits the percentage of the camphorquinone-based photoinitiator system, and the percentage and composition of the photoinitiator component for RelyX ARC is unknown. The degree of conversion of RelyX ARC was not influenced by either the curing light or chemical catalyst (54.26% to 68.68%); however, Panavia F just reached an adequate monomer conversion rate (44.9% and 54.1%) when QTH light was used, regardless of the incorporation of ED Primer. The results suggest that RelyX ARC may contain camphorquinone in a higher percentage than Panavia F, since the curing units with a narrower spectrum range for the absorption peak of this photoinitiator (the LED and the argon ion laser) did not influence monomer conversion, compared to the QTH curing light. Therefore, Panavia F, when cured with the LED and the laser without ED Primer incorporation, reached a poor degree of conversion in comparison with that achieved with the QTH light. Thus, ED Primer should be incorporated with Panavia F in any manipulation procedure in order to achieve a satisfactory degree of monomer conversion and, consequently, clinical success.

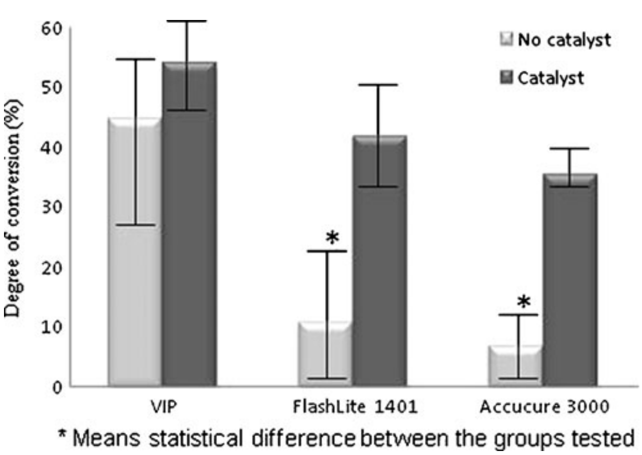

Fig. 2 Degrees of conversion (means±SD) for Panavia F (*$p<0.05$)

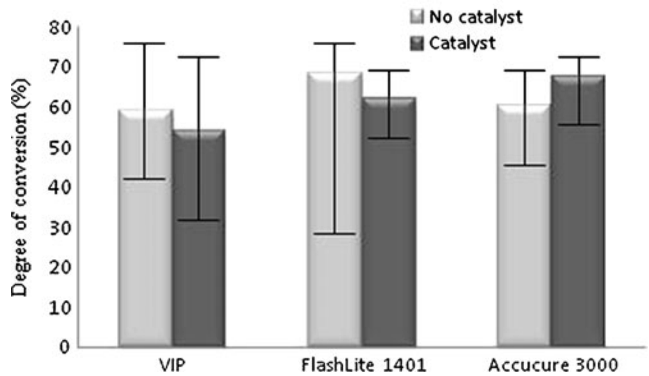

Fig. 3 Degrees of conversion (means±SD) for RelyX ARC

Faria-e-Silva et al. [2] found that the degree of conversion of Panavia F was approximately 60% when photocured with halogen light under a resin composite disk. In this study, the highest degree of conversion of Panavia F was 54.1% when used with the chemical catalyst and cured with the QTH light. However, other authors [30] have stated that when cured directly with a LED, this cement reaches about 48% monomer conversion; however, in this study when this cement was cured with the LED unit under a resin composite disk, the degree of conversion reached about 10.91%, which is unsatisfactory for clinical applications.

The curing units used in this work emitted different irradiances and final energy densities after 40 s for photoactivation was adopted for all light sources (QTH and laser, 600 mW/cm^2 and 24 J; LED, 1,200 mW/cm^2 and 48 J). Although higher values of irradiance induce greater heat generation, the composite disk attenuated the temperature alteration caused by the LED since the composite had a low thermal conductivity [27, 29], and did not affect the degree of conversion of the tested resin cements. The curing unit energy density did not influence the degree of conversion of Panavia F cured without ED primer, since QTH provided a significantly higher degree of conversion than the LED when ED Primer was not used. This finding may be associated with the photoinitiator content of Panavia F and the broad spectral range of QTH light that can reach alternative photoinitiators better than the second-generation LED used in this study, even though the latter emits a higher irradiance for the same curing time than the QTH light. For conventional camphorquinone-based resin composites, no difference has been observed in the development of physical properties, such as the degree of conversion, when LED and QTH units are compared [13, 31].

The argon ion laser has been described as a promising light source for curing resinous materials [21]. In spite of some benefits, such as an economic benefit of 25–33%, adequate depth of cure, and enhancement in resin physical properties after polymerization, the laser absorption peak is at 488 nm, displacing the camphorquinone absorption peak at 468 nm [32, 33]. In some situations, this distance can make activation with the argon ion laser inefficient [21, 34]. In this study, the argon ion laser, working with the same energy density as the QTH light, achieved an inadequate degree of conversion of Panavia F alone, due to the offset narrower spectral peak at about 488 nm, which could not initiate the photoinitiators present in this cement under the resin composite disk. When used with ED Primer, Panavia F cured with argon ion laser did not display a significantly different degree of monomer conversion than that cured with the QTH or LED. For RelyX ARC there was no difference in the degree of conversion between the curing lights, indicating that the argon ion laser is suitable for cementing of indirect restorations, similar to the QTH and LED lights, when this material is used.

For the photoactivation procedure, a resin composite disk was prepared to simulate an indirect restoration, which is able to provide adequate physical and mechanical cement properties [17]. It is important to simulate a clinical situation representative of cementing an indirect restoration, since light would be attenuated by the composite [2, 5] or ceramic [17, 26, 35, 36], and the degree of cure might be lower than with direct curing. Besides, the resin luting thickness was 0.5 mm, which is close to that seen in clinical situations, and it allowed satisfactory measurement by FTIR [1, 5]. Some studies [26, 30] used specimens with a cement thickness of about 1–2 mm, which is not clinically acceptable, as it may result in a lower degree of conversion at the bottom of the sample due to the depth of cure of the resinous material [21, 30] and differences in the light curing irradiance that reaches all surfaces of the composite [14, 37].

The resin cements were activated totally by dual curing. The chemical curing of the luting materials with incorporation of the chemical catalyst was not tested because the results of some studies have shown that chemical curing alone does not promote an adequate degree of conversion of resin cements [2, 5]. On the other hand, many manufacturers state that resin cement photoactivation may be delayed for 5 or 10 minutes, since important chemical curing can occur and such a delay does not stop the conversion of monomers by the free radical polymerization center [2]. In this study specimens were immediately photocured for the sake of standardization because the manufacturers of some resin luting cements do not recommend such a delay in photoactivation.

The chemical catalysts used in this study contained benzoyl peroxide, which might be able to start some chemical cure at the material matrix [5]. The self-cure is initiated with a reaction between benzoyl peroxide and tertiary amine that releases free radicals which promote monomer conversion regardless of exposure to light curing [38]. Thus, the catalyst of the Scotchbond Multi-Purpose system contains only benzoyl peroxide as an initiator system. This catalyst did not influence the degree of cure of RelyX ARC, regardless of the light curing source. This may be explained by the chemical reaction that normally occurs between benzoyl peroxide and the tertiary amine present in the composite material. If the tertiary amine supply is lower than the benzoyl peroxide content, the reaction cannot initiate, and the degree of conversion remains undisturbed [38]. However, ED Primer is a system that contains a tertiary amine/benzoyl peroxide system, providing a complete self-cure reaction that could have improved the physical properties of Panavia F when mixed with this resin cement [2]. For Panavia F, a primer A and B mixture incorporated with the cement increased the degree of conversion when the LED and the argon ion laser were used; whereas a similar degree of conversion was obtained when the QTH curing light was used. So for Panavia F, the ED Primer is essential for maintaining a satisfactory degree of monomer conversion when cured with the light sources most frequently used in clinical practice.

For indirect esthetic restorations, light plays an important role in promoting satisfactory conversion of the resin cement, ensuring adequate cementation [2, 5]. However, the attenuation of the irradiance by ceramic or resin indirect restorations with a 2-mm thickness is such that only a half of the irradiance reaches the resin cement beneath these materials [5, 39]. For metallic crowns, the chemical curing and incorporation of the catalyst into the resin luting cement might compensate for the total attenuation of the curing light, since that would cause an effect only around the margin. Thus, clinicians should incorporate the chemical catalyst into the manipulation of resin cements, since it improves or does not affect the degree of conversion of these luting materials.

Conclusion

Based on these results, it can be concluded that the argon ion laser, LED, and QTH light promote an adequate degree of conversion for RelyX ARC, regardless of the addition of the chemical catalyst. Nevertheless, Panavia F, when photocured with the LED or argon ion laser, should be mixed with the chemical catalyst (ED Primer) to achieve a satisfactory degree of conversion, especially in situations affected by the attenuation of light. Thus, Panavia F should be used with the incorporation of the ED Primer on manipulation and photocured with a QTH light unit.

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