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Original

Panavia F: the role of the primer

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Abstract: This study evaluated the rate of polymerization (R_p) and degree of conversion (DC) of Panavia F when self- or dual-activated, and the influence of either using or not using a primer containing co-initiators (ED Primer) mixed with the material. The conversion reaction was monitored using real-time infrared spectroscopy with an attenuated total reflectance device. The cement was mixed, put onto the device and irradiated for 20 s (dual-cured). A self-cured group was also prepared. Similarly, dual- and self-cured groups were evaluated after mixing ED Primer with the cement. The DC was monitored for 1 h and the R_p was calculated. For the dual-cured mode, the most significant effect of ED Primer was to reduce the R_p^{max}. For the self-cured mode, the primer was shown to be essential for polymerization of Panavia F: without ED Primer, the reaction started only after approximately 500 s, with a final DC of 50%, whereas a continuous increase in conversion was observed for the group mixed with ED Primer, with a final DC of 74%. The ED primer is essential for proper polymerization of Panavia F; the cement becomes independent of the light when the primer is used. (J Oral Sci 51, 255-259, 2009)

Keywords: degree of conversion; light-activation; polymerization; resin cement; rate of polymerization; self-cure.

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Introduction

Resin-based luting agents are commonly used in dentistry. During the cementation of indirect restorations and intra-radicular posts using resin cements, proper polymerization of the luting material is essential for clinical success of the restorations (1,2). Low values for degree of conversion (DC) might reduce the retention of the indirect material or the post (3) and contribute to restoration failures.

Dual-cured resin cements were introduced to combine the favorable characteristics of self- and light-cured agents. The rationale was to develop a material with extended working time capable of reaching high DC in either the presence or absence of light (4). During the cementation of posts and thick indirect restorations, exposed marginal areas can benefit largely from the photo-activation, as they are readily accessible to the curing light; however, a significant reduction in its intensity might occur due to reflecting and scattering effects. Therefore, in some situations, the polymerization reaction may be mainly activated by a chemical mechanism (self-cure). However, it has been shown that dual-polymerized resin cements might depend on being exposed to light to achieve better mechanical properties (5).

One of the most widely-used commercial dual-cured resin cements is Panavia F (Kuraray Co., Osaka, Japan). This system consists of two pastes and a self-etching primer (ED Primer) containing co-initiators, and it is recommended that the primer should be applied to the substrate before cementation. Until the present time, the effect of ED Primer on the properties of Panavia F had not been clarified. Studies evaluating the polymerization and mechanical properties of this cement usually do not use the primer. For instance, Kumbuloglu et al. (6) observed that Panavia F is extremely dependent on light exposure to obtain better mechanical properties, and Garcia et al. (7) detected lower bond strength to dentin when the cement was not light-cured; however, these authors did not use ED Primer.

On the other hand, on using ED Primer, Piwowarczyk et al. (8) found that Panavia F presented higher bond strength after thermal cycles and water storage when the material was self-activated. It seems reasonable to think that the previously mentioned results (7,8) are related to the non-application of ED Primer before luting, and therefore it is important to evaluate its effect on the luting material.

The aim of this study was to evaluate the DC and the rate of polymerization (R_p) of Panavia F when self- or dual-activated, and the influence of either using or not using the primer containing co-initiators mixed with the material. The working hypothesis was that the polymerization process of the dual-cured material would not be dependent on light exposure provided that the primer containing co-initiator is applied.

Materials and Methods

The dual-cured resin cement Panavia F and the self-etching primer ED Primer were tested. The composition of the materials is shown in Table 1. The DC of the luting agent was measured using a Fourier transform infrared spectrometer (Prestige21; Shimadzu, Columbia, MD, USA) equipped with an attenuated total reflectance device composed of a horizontal ZnSe crystal, with a 45° mirror angle (Pike Technologies; Madison, WI, USA). Equal amounts (27 mg) of base and catalyst pastes were weighed on an analytical balance, mixed for 15 s and placed onto the crystal. The material was covered with a Mylar strip to avoid polymerization inhibition by contact with oxygen.

A disc (2 mm thick × 10 mm in diameter) of resin composite (Filtek Z250; 3M ESPE, St. Paul, MN, USA), shade A2, was prepared to simulate an overlaying laboratory-processed composite restoration. The disc was placed over the Mylar strip and light-activation was carried out through the composite for 20 s, with the light guide tip of the curing unit (Optilux501; Demetron Kerr, Orange, CA, USA – 600 mW/cm^2) placed in contact with the disc. Additionally, a self-activated group was obtained by shielding the luting material from the curing light.

In order to evaluate the impact of ED primer on the polymerization of Panavia F, 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, 0.1 µL (1.4 mg) of this solution was dispensed onto a glass slab using a micropipette (model NPX2; Nichipet EX, Santa Clara, CA, USA) and mixed with the base and catalyst pastes of the dual cement for 15 s. The volume of ED Primer was determined after pilot experiments: after mixing the primer with the resin cement, the mixture had a similar aliphatic-to-aromatic C=C peak ratio as that of the cement agent.

To measure the DC, the IR Solution software (Shimadzu) was used in the monitoring scan mode with Happ-Genzel apodization, for collecting spectra in the 1,665 to 1,580 cm^{-1} range, with resolution of 4 cm^{-1}. With this setup, one

Table 1 Materials used in the study

Material	Composition*	Batch #
Panavia F	*Paste A*: 10-methacryloyloxydecyl hydrogen phosphate (MDP), hydrophobic and hydrophilic dimethacrylate, benzoyl peroxide, camphoroquinone, colloidal silica	00245F
	Paste B: sodium fluoride, hydrophobic and hydrophilic dimethacrylate, diethanol-*p*-toluidine, T-isopropylic benzenic sodium sulfinate, barium glass, titanium dioxide, colloidal silica	00024B
ED Primer	*Primer A*: 2-hydroxyethyl methacrylate (HEMA), MDP, NM- aminosalicylic acid, diethanol-*p*-toluidine, water	00207E
	Primer B: NM- aminosalicylic acid, T-isopropylic benzenic sodium sulfinate, diethanol-*p*-toluidine, water	00086E

*Information provided by the manufacturer.

scan was acquired every 6 s during polymerization. All analyses were performed under controlled temperature (23 ± 2°C) and humidity (60 ± 5%) conditions. The DC was calculated using a baseline technique (9), considering the intensity of aliphatic C=C stretching vibration (peak height) at 1,635 cm^{-1} and using the symmetric ring stretching at 1,608 cm^{-1} as an internal standard, from both polymerized and unpolymerized samples.

DC for each scan was determined by subtracting the percentage of remaining aliphatic C=C from 100%. Conversion vs. time data was plotted and Hill's three-parameter non-linear regressions were used for curve fitting. As the coefficient of determination was greater than 0.9 for all curves, the R_p (%.s^{-1}) was calculated using the data resulting from the non-linear regressions, and the maximum R_p (R_p^{max}) was recorded.

Results

The DC vs. time curves are shown in Fig. 1. Logarithmic time scale was used for a better comparison of the tested conditions. For the dual-cured mode without using ED Primer, a rapid increase in DC was found in the first 20 s of the reaction. After this period, the reaction speed decreased, reaching a plateau at 100 s, when the DC was around 60%. Without light-activation (self-cured mode) and primer, the polymerization reaction only started after approximately 500 s, and a continuous increase in DC until 2500 s was found. However, conversion stabilized with a DC of only 50%.

With regard to the impact of ED Primer on the R_p, a rapid increase in DC in the first 60 s was found for the dual-cured mode. However, as shown in Fig. 2, the R_p^{max} for this group (5 s) was lower than that for the dual-cured mode without ED Primer (6 s). After R_p^{max}, a reduction in the speed of polymerization reaction was observed, but a continuous increase in conversion was observed until a final DC of approximately 74% was reached (Fig. 1). This value was higher than that observed for the dual-cured group without ED Primer (64%).

For the self-cured mode, the use of ED Primer promoted a continuous increase in conversion during the first 500 s, when the cement reached a DC of around 60% (Figure 1). In addition, this group had a DC of approximately 75% after 1 h. As shown in Fig. 2, the R_p^{max} was also higher than the values obtained without ED Primer, and the peak in the polymerization rate curve was displaced to the left (around 1100 s for self-cured mode without ED Primer vs. 100 s for self-cured mode with ED Primer).

Discussion

The results of the present study indicated that the use of a primer containing co-initiators is essential for the polymerization of resin luting agent when it is not exposed to polymerizing light. The manufacturer classifies Panavia F as a dual-cure resin cement, that is, the material should be polymerized by both chemical and light activation. The photoinitiator system of the luting agent is based on camphoroquinone, which absorbs energy when it is exposed to visible light in the 400 to 500 nm wavelength range, and reacts with tertiary amine to form an excited state complex that breaks down into reactive free radicals (10). On the other hand, self activation is obtained through the reaction between benzoyl peroxide and tertiary amine, generating free radicals that will break the aliphatic C=C and initiate

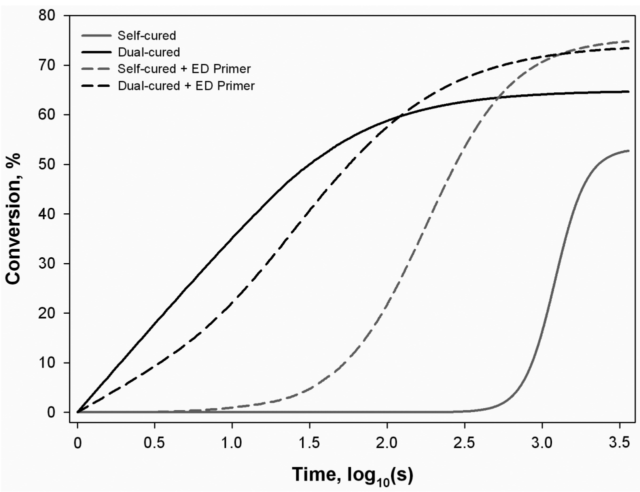

Fig. 1 Progress of polymerization of Panavia F conversion for the four tested conditions.

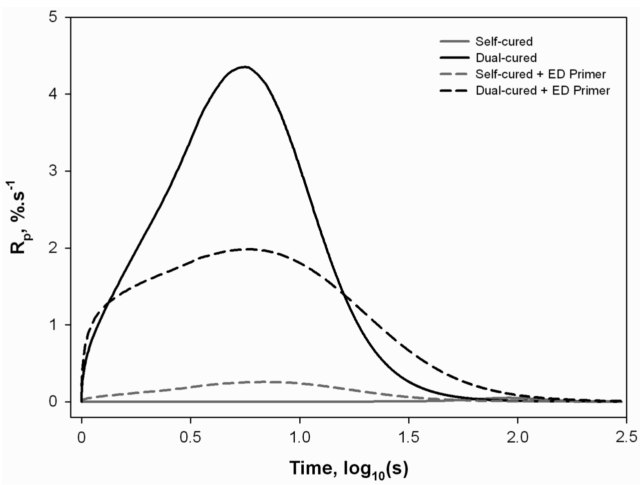

Fig. 2 Variation of rate of reaction of Panavia F for the four tested conditions (slopes of curves of Fig. 1).

the polymerization process (11).

The slower reaction and the reduced DC of self-cured Panavia F when not mixed with ED Primer might be the result of a low amount of tertiary amine (diethanol-*p*-toluidine) in the catalyst paste (11). The rationale is that the mixture of the primer and cement may increase the amount of amine, improving the polymerization reaction, and increasing the final DC. In addition, the primer is much less viscous than the catalyst paste of the cement, and therefore the mixture of the co-initiators within the bulk of the material is probably better when the primer is applied. Another potential explanation for the beneficial impact of ED Primer is that it contains T-isopropylic benzenic sodium sulfinate as a co-initiator in primer liquid B. This salt might react with the acid resin monomers present in primer A, and with the resin cement itself, to produce free-radicals that would enhance the polymerization reaction (12,13).

Most manufacturers recommend that clinicians should wait at least 5 min before finishing and polishing a recently luted restoration, as they would generally be placed under occlusal loads approximately 10 min after the luting procedure. In the present study, after 5 min, the luting agent had a DC higher than 60% in the dual-cured mode, irrespective of whether or not ED Primer was used. However, a 2-mm-thick resin composite was used to simulate indirect light-activation, and it should be considered that the light reaching the cement layer might vary as a function of different curing devices and different thicknesses, shades or opacities of the intervening restorative.

On the other hand, without light-activation and without the primer, no significant conversion was found after 5 or 10 min, and it is assumed that this material would not withstand the occlusal loading or even the finishing procedures in clinical practice. Nonetheless, the DC increased to 52 and 65% at 5 and 10 min, respectively when the ED Primer was used. These results again indicate the importance of ED Primer for proper polymerization of Panavia F; the luting material becomes independent of the light when the primer is used. In other words, the luting material achieves proper polymerization when the ED Primer is mixed with the cement, even when no light-activation is carried out. However, it has been shown that luting materials might be dependent on being exposed to light to achieve better properties (5). Therefore, further evaluation of mechanical properties is required.

When Panavia was mixed with the primer and light-activated, a reduction in the R_p^{max} was found in spite of the increased final DC. This reduction might be related to the presence of solvent in the primer, interfering in and slowing down the polymerization process. Indeed, the polymerization reaction occurred faster when the cement was light-activated without using the primer. Nevertheless, reducing the R_p^{max} might be beneficial in clinical practice, as reduced rates of polymerization are generally linked with lower shrinkage stress development (14).

However, the reduction in the polymerization rate might also be associated with the formation of fewer polymer growth centers, which could favor the formation of loosely cross-linked polymers, with poorer mechanical properties (15). Nonetheless, it is difficult to predict whether alteration in the R_p could alter the polymer network formation. Moreover, although no additional increase in DC after 1h was observed for any group in the present study, longer post-curing periods and the development of mechanical properties should be evaluated in further studies.

According to the present results, the use of a primer containing accelerators and co-initiators was advantageous for increasing the DC and R_p of the dual-cured resin cement system tested here; the use of the primer would potentially increase the conversion of double bonds in regions with compromised access to light. However, this beneficial effect would not occur in procedures such as the resin-coating technique (16), where the adhesive system is covered with low-viscosity composite to produce a tight seal that would reduce pulp irritation and post-operative sensitivity. Therefore, the use of resin cement after the self-etching protocol recommended by the manufacturer would be preferable, due to improvement in DC.

It is also important to emphasize that there is a limitation involving the R_p data for light-cured samples, since one scan was acquired every 6 s, and the photo-polymerization reaction is very fast. Moreover, the effect of any primer containing co-initiators may occur mainly at the interface between the resin cement and the primer layer, and thus only a few micrometres thickness of the cement may undergo increase in DC. The remaining material would poorly polymerize when not exposed to light. The mixture of the primer and luting materials would overcome this problem, but care should be taken to use a low proportion of ED Primer in order not to decrease the working time of the Panavia F drastically, and impair the luting procedure.

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