

Impact of immediate and delayed light activation on self-polymerization of dual-cured dental resin luting agents

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Abstract

This study investigated the impact of immediate and delayed light activation on self-polymerization of a model dual-cured luting agent. The material presented the following components: base paste – 2,2-bis[4-(2-hydroxy-3-methacryloxyprop-1-oxy)phenyl]propane/triethylene glycol dimethacrylate (TEGDMA), camphorquinone, dimethyl-*p*-toluidine, butylated hydroxytoluene (BHT), glass fillers; catalyst paste – bisphenol-A ethoxylated dimethacrylate/TEGDMA, benzoyl peroxide, BHT, fillers. The pastes were mixed and seven polymerization scenarios tested: immediate light activation using low (5 J cm^{-2}) or high (20 J cm^{-2}) energy dose; delayed light activation (after 2 min – short delay) using low or high dose; delayed light activation (after 10 min – long delay) using low or high dose; and self-polymerization only. The degree of conversion (DC) and rate of polymerization (R_p) were evaluated for 30 min by real-time infrared spectroscopy. The lowest DC was detected for the self-polymerized and immediate–low dose groups, whereas the immediate–high dose and short delay–high dose groups showed the highest values. For the self-polymerized and immediate–high dose samples, R_p^{max} was detected after approximately 7 s, whereas this took approximately 14 s for the immediate–low dose group. R_p^{max} for the immediate–high dose group was higher than for the self-polymerized sample, which in turn was higher than for the immediate–low dose group. R_p^{max} for the short delay groups was higher than for the long delay groups. In conclusion, the extent of self-polymerization was influenced by the light dose reaching the material, which was dependent on high radiant exposure for optimal polymerization and the moment at which the light was applied; the short delay increased the DC for lower doses, while also generally decreasing the R_p for all scenarios.

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1. Introduction

Resin-based luting agents are increasingly being used in dentistry for luting ceramic restorations and intra-radicular posts [1]. In order to obtain high bond strengths after cementation, adequate light energy is required to reach the luting material to ensure optimal polymerization [2]. Dual-cured materials were introduced to conciliate the favorable characteristics of self- and light-cured agents,

that is, a material with an extended working time capable of reaching proper polymerization in either the presence or absence of light. However, in clinical situations, such as the cementation of thick ceramic restorations, significant light attenuation might occur [3–7]. In these conditions, it is uncertain to what extent each type of curing reaction will occur, or what period of time is required for proper polymerization of dual-cured materials.

Previous studies have assessed the properties of dual-cured resin luting agents. Luting agents that were only self-activated were found by some investigators to not be comparable to those that were light-activated [3–6,8–10].

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While others have shown that the chemical curing effect might be material-dependent [11,12], the impact of the light activation on the self-polymerization mechanism of dual-cured materials has not been well established. For instance, it is unknown whether the self-curing might be affected by the light energy dose reaching the luting material, or whether it might be influenced by the moment at which light is applied.

It has been speculated that a delay in the light activation of dual-cured materials would enhance their properties [13], as the immediate exposure to light and formation of cross-linked polymer chains could interfere with the self-curing by entrapping polymerization promoters and unreacted monomers into the network, therefore interfering with the self-cure mechanism. However, this effect has not yet been recognized and warrants further investigation.

The purpose of this study was to investigate the impact of immediate and delayed light activation on the self-polymerization of a model dual-cured resin luting agent, with immediate light-activated and self-polymerized samples as control groups. The hypotheses tested were: (i) the self-polymerization would be dependent on the light energy dose reaching the material; and (ii) the delayed light activation would have no significant effect on the polymerization reaction of the resin luting agent.

2. Materials and methods

2.1. Purification of monomers

Before formulating the model material to be tested, the monomers were purified to remove inhibitors and impurities. The reagents were dissolved in dichloromethane, then washed three times with 10 wt.% aqueous NaOH solution, three times with 1 wt.% aqueous HCl solution and once with saturated aqueous NaCl solution. The water was removed by drying with anhydrous CaCl₂ salt; the solution was then filtered and the dichloromethane removed by distillation under vacuum using a rotary evaporator.

2.2. Formulation of the model material

The model dual-cured resin luting agent was formulated using the purified monomers 2,2-bis[4-(2-hydroxy-3-methacryloxyprop-1-oxyl)phenyl]propane (Bis-GMA), bisphenol-A ethoxylated dimethacrylate (Bis-EMA) and triethylene glycol dimethacrylate (TEGDMA), all from Esstech Inc. (Essington, PA, USA). Two resin mixtures were obtained and loaded with a mass fraction of 60% of 0.4 μm silanated barium borosilicate glass fillers (Esstech Inc.), with the following components (mass fraction):

- *base paste*: Bis-GMA:TEGDMA (1:1 ratio) comonomer, camphorquinone (0.8%) as photosensitizer, dimethyl-*p*-toluidine (2.5%) as coinitiator and butylated hydroxytoluene (BHT; 0.2%) as radical scavenger;

- *catalyst paste*: Bis-EMA:TEGDMA (1:1 ratio) comonomer, benzoyl peroxide (2.5%) as self-polymerization promoter and BHT (0.2%).

In order to simulate the actual extent of the self-polymerization achieved by dual-cured dental luting agents [5], the amount of polymerization promoters and inhibitor were adjusted after pilot studies using commercial dual-cured agents. The formulation corresponds to a similar working time to the proprietary materials.

2.3. Real-time infrared spectroscopy

The real-time polymerization of the model material was evaluated by Fourier transform infrared (FTIR) spectroscopy (Prestige21; Shimadzu, Columbia, MD, USA), using an attenuated total reflectance (ATR) device composed of a horizontal ZnSe crystal. Fig. 1 shows the experimental setup used in this study. Equal volumes of base and catalyst pastes were mixed for 15 s, placed onto the crystal and covered with a transparent polyester strip. The thickness of the luting agent film was approximately 50 μm, which is relevant to a cement layer thickness in clinical luting procedures [14]. The use of a thin film is important to avoid reduction in light penetration within the bulk of the luting material during photoactivation. The following polymerization scenarios were tested:

- *immediate light activation using low or high energy dose*: the material was immediately light-activated for 10 or 40 s using a quartz–tungsten–halogen curing unit (XL3000; 3M ESPE, St. Paul, MN, USA, 500 mW cm⁻²), applying an energy dose of 5 or 20 J cm⁻²;
- *delayed light activation (short delay) using low or high energy dose*: the luting agent was light-activated for 10 or 40 s 2 min after placing the material onto the crystal, applying an energy dose of 5 or 20 J cm⁻²;
- *delayed light activation (long delay) using low or high energy dose*: the luting agent was light-activated for 10 or 40 s 10 min after placing the material onto the crystal, applying an energy dose of 5 or 20 J cm⁻²;
- *self-polymerization*: no light activation procedure was performed; polymerization relied upon self-activation only.

The material was shielded from the ambient light before and after the light activation. The polymerization reaction was monitored in the absorbance (Abs) mode in real-time for 30 min using Happ–Genzel apodization. Monitoring started immediately after the mixed material was placed on the ATR crystal, collecting spectra in the 1680–1540 cm⁻¹ range, with a resolution of 8 cm⁻¹. With this setup, one scan every second was acquired. For each polymerization scenario, five specimens were tested under controlled temperature (25 ± 1 °C) and humidity (60 ± 5%) conditions. The degree of conversion (DC) for each scan

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