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Stability of initiation systems in acidic photopolymerizable dental material

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ABSTRACT

Objectives. This study simulated the shelf life to evaluate the stability of initiation systems on acidic photopolymerizable dental material, through an experimental self-adhering flowable composite resin (SACR).

Methods. An SACR model was formulated with monomers Bis-GMA, TEGDMA, acidic monomer (GDMA-P), and inorganic fillers. Initiation system combinations of camphorquinone (CQ), tertiary amine (EDAB), diphenyl phosphine oxide (TPO), phenylbis phosphine oxide (BAPO), and the diphenyliodonium hexafluorophosphate (DPIHFP) were tested. Five SACRs were evaluated, varying the initiation system: SACR_{CQ}, SACR_{CQ+EDAB}, SACR_{CQ+EDAB+DPIHFP}, SACR_{BAPO} and SACR_{TPO}. The SACR were stored at 23 °C and, according to shelf life, were evaluated for degree of conversion (DC), polymerization rate (Rp) and microtensile bond strength (μ TBS). The DC was evaluated after 0, 1, 2, 4, 8, 12, 24 and 48 storage weeks of SACRs. Bonding to dentin was performed after 0, 4, 8, 12 and 24 storage weeks of SACRs and tested immediately (24 h) and after 6 and 12 months by μ TBS. Halogen light curing unit (3M ESPE) was used for photoactivation procedures.

Results. SACR_{CQ+EDAB+DPIHFP} showed higher Rp. The DC of SACR_{CQ+EDAB}, SACR_{CQ+EDAB+DPIHFP}, and SACR_{BAPO} were similar (40%) and higher than SACR_{TPO} and SACR_{CQ} (20 and 10%, respectively), staying stable up to 48 weeks. The SACR_{CQ}, SACR_{CQ+EDAB}, and SACR_{TPO} had pre-testing failure on μ TBS. The shelf life of SACRs did not affect the μ TBS of the groups that obtained adhesion. Furthermore, the immediate μ TBS (MPa) SACR_{CQ+EDAB+DPIHFP} (8.4) was similar to SACR_{BAPO} (10.5); however, after 12 months, only SACR_{CQ+EDAB+DPIHFP} maintained the μ TBS.

Significance. The choice of a suitable initiation system is crucial to the performance and stability of acidic photopolymerizable dental material.

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1. Introduction

Self-etch/adhesive resin or self-adhering materials were developed in dentistry to simplify the restorative procedure

by reducing the number of bonding steps, the application time, and technique sensitivity for the operator [1]. This class of dental materials can be commercially found as one-bottle self-etch adhesive systems, self-adhesive resin cements, or as

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a more recent product, the self-adhering flowable composite resin [2–4].

The approach of self-adhering materials has been claimed to be more user friendly; however, mixing all ingredients into one bottle has caused some problems for these materials regarding phase separation, reduced rate of polymerization, and reduced shelf life [5–8]. These should be attributed to the difficulty of obtaining stable materials in the complex mixture of monomers (monofunctional, crosslinking, and strongly acidic ones), inorganic fillers, solvents, and photoinitiator systems. The most commonly used photoinitiator system in dental polymers is based on the visible-light photosensitizer camphorquinone (CQ), a Norrish type II photoinitiator. It requires a co-initiator, such as the tertiary amine, which donates hydrogen to promote an efficient polymerization reaction [9]. However, some studies have demonstrated that the tertiary amine is chemically unstable in acidic materials [10–15], as with the self-adhesive ones and the all-in-one materials. In addition, it was suggested that the acid–base reaction of the acidic monomers with tertiary amines in the materials that are chemical polymerizable can decrease the shelf life stability of these materials and may affect the polymerization reactivity and adhesion process [12].

Since then, some studies have proposed using unitary initiation systems that are co-initiator-free, such as a Norrish type I photoinitiator; or CQ-based binary initiation systems with alternative co-initiators as amine substitutes; or CQ-based ternary initiation systems would also present better color stability, degree of conversion, rate of polymerization, and mechanical properties [9,16–21] and would have shelf life stability. Today, the self-adhering materials are completely, or at least in part, photosensitive; hence, it is important to evaluate shelf life stability and its influence on polymerization reaction, adhesion process, and the mechanical properties of the CQ/amine-based materials in a visible-light polymerization reaction.

Despite the possible effects of the initiation system added as a constituent of acidic materials, there is no evidence in the literature regarding the shelf life of this component on the formulation stability and properties of acidic photopolymerizable dental material, such as self-adhering flowable composite resin (SACR). Therefore, the aim of this study was to evaluate the influence of shelf life on a model SACR, in terms of its polymerization reaction and bonding effectiveness to dentin, when different initiation systems are added to this visible-light curing acidic material. The hypotheses tested were that the initiation systems are unstable in acidic photopolymerizable dental material and that the shelf life affects the SACR properties.

2. Materials and methods

2.1. Reagents

The bisphenol A glycidyl dimethacrylate (Bis-GMA; Esstech Inc., Essington, PA, USA) and triethylene glycol dimethacrylate (TEGDMA; Esstech), were used as received. The acid phosphate monomer 1,3 glycerol dimethacrylate phosphate (GDMA-P) was synthesized as previously described

[22]. Silanized inorganic particles (barium borosilicate glass, 0.7 μm average size and 9% silane) were used as fillers (Esstech). The initiators camphorquinone (CQ; Esstech), ethyl 4-dimethylaminobenzoate (EDAB; Aldrich Chemical Co., Milwaukee, WI, USA), diphenyliodonium hexafluorophosphate (DPIHFP; Aldrich Chemical), diphenyl (2,4,6-trimethylbenzoyl)-phosphine oxide (TPO; Aldrich Chemical), and phenylbis (2,4,6-trimethylbenzoyl)-phosphine oxide (BAPO; Aldrich Chemical) were used to render the materials photocurable.

2.2. Formulation of the experimental groups

A model SACR was prepared through the intensive mixture of 30 wt% of Bis-GMA, 10 wt% TEGDMA, 20 wt% acidic phosphate monomer GDMA-P, and 40 wt% inorganic fillers.

An optimal photoinitiators and co-initiators concentration of 0.5 mol% CQ [23], 1 mol% EDAB [23], 1 mol% DPIHFP [9], 1 mol% TPO [24], and 1 mol% BAPO [25] were established from the standpoint of degree of conversion according to previous studies and were added to the model SACR according to the monomer moles. Five SACR were evaluated, varying the initiation system: SACR_{CQ}, SACR_{CQ+EDAB}, SACR_{CQ+EDAB+DPIHFP}, SACR_{BAPO}, and SACR_{TPO}. The structural formula and the molecular weight of the initiators evaluated are presented in Table 1. No radical scavenger was added to avoid interference with the polymerization kinetics. Photoactivation procedures were carried out using a halogen light curing unit (XL 3000; 3M ESPE, St. Paul, MN, USA) with 400 mW/cm² irradiance confirmed by a digital power meter (Ophir Optronics, Danvers, MA, USA).

2.3. Evaluation of SACR stability and storage time conditions

To evaluate the stability of initiator system on SACR in long term, the shelf life was simulated. After formulation the SACRs were stored in the incubator at 23 °C and, according to the storage time, were evaluated for degree of conversion (DC), rate of polymerization (Rp), and immediate and longitudinal microtensile bond strength (μTBS) following the procedures described below.

Analysis of Rp was performed immediately after SACRs formulation. The DC was evaluated after 0, 1, 2, 4, 8, 12, 24, and 48 storage weeks of SACRs. Bonding to dentin was performed after 0, 4, 8, 12, and 24 storage weeks of SACRs and tested by μTBS after 24 h and 6 and 12 months.

2.4. Rate of polymerization and degree of conversion

The reaction of polymerization of the SACR ($n=3$) was evaluated using real-time Fourier transform mid-infrared (FTIR) spectroscopy (Prestige 21; Shimadzu, Tokyo, Japan) with an attenuated total reflectance device (ZnSe crystal). The SACR ($\sim 3 \mu\text{l}$) was directly dispensed on the ZnSe crystal.

To DC and Rp evaluation, a reading for the material was taken using setup monitoring scan mode by IRsolution software (SHIMADZU, Columbia, MD, USA), 1 scan per second and 4 cm⁻¹ resolution. The sample scanning was performed simultaneously with the photoactivation for 60 s. The DC (%) and

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