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# Influence of photoinitiator system and nanofiller size on the optical properties and cure efficiency of model composites

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## ABSTRACT

**Objective.** To establish the relationship between photoinitiator system and nanofiller size on the optical properties and cure efficiency of model composites.

**Methods.** Model composites based on BisGMA/TEGDMA (60:40 mol%) were loaded with 40 wt% of 7 nm or 16 nm-sized filler particles. One of the following photoinitiator systems was added: camphorquinone (CQ) associated with an amine (EDMAB), monoacylphosphine oxide (TPO), or bisacylphosphine oxide (BAPO). The optical properties of disk-shaped specimens were measured 24 h after curing and repeated after storage in water for 90 days and coffee for 15 days. A large spectrum LED unit (Bluephase G2, Ivoclar Vivadent) was used for photoactivation. CIE  $L^*a^*b^*$  parameters, color difference ( $\Delta E$ ), and translucency parameter (TP) were calculated. Knoop hardness readings were taken at top and bottom composite surfaces. Cure efficiency was determined by bottom/top hardness ratio. Data were statistically analyzed at  $\alpha = 0.05$  significance level.

**Results.** Composites formulated with 16 nm particles had higher CIE  $L^*$  than those with 7 nm particles in all storage conditions. BAPO-based composites generally had lower CIE  $a^*$  than the other composites. The group TPO + 16 nm before storage and all groups with 16 nm-sized particles after storage had lower CIE  $b^*$  (i.e. lower degree of yellowing) than the other groups. TPO-based materials had higher color stability. The cure efficiency was not significantly affected by photoinitiator system or particle size. CQ + 7 nm had the lowest and BAPO + 16 nm the highest hardness values.

**Significance.** Combination of photoinitiator system and filler particle size might affect the optical properties of composites, with low influence on cure efficiency.

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## 1. Introduction

The photoinitiator system in dental composites is a group of molecules capable of absorbing light and as a result, directly or indirectly, giving rise to reactive species that can initiate polymerization. Photoinitiator molecules usually have in their structure a carbonyl group, which presents one electron capable of being transformed in anti-bonding orbital when exposed to light at appropriate wavelengths [1]. Association of camphorquinone (CQ) with a tertiary amine has been largely used as the photoinitiator system since the introduction of visible-light activated dental composites. CQ is a solid chromophore, meaning that the molecular structure includes a chromatic group that makes the material photoactive, in turn bringing a strong yellow color for the material. For esthetic reasons, the use of photoinitiator systems alternative to CQ/amine has been suggested [2].

The use of less yellow photoinitiators such as the phosphine oxide derivatives monoacylphosphine oxide (MAPO, also known as TPO) or bisacylphosphine oxide (BAPO) could minimize problems related to the color instability of CQ-based materials. TPO and BAPO absorb light at shorter wavelengths than CQ and, as a consequence, present a very pale yellow color or no color at all. BAPO and TPO are Norrish Type 1 photoinitiators, which means that they generate free-radicals by a photocleavage process that does not require a co-initiator. Type 2 photoinitiators, such as CQ, need a co-initiator molecule to effectively generate free-radicals, which are formed from displacement of hydrogen from the photoinitiator [2]. Amines are known to generate by-products during photoreaction, which tend to cause discoloration from yellow to brown, depending on the type and fraction of amine in the system [3,4]. In addition, there is a potential cytotoxicity of unreacted CQ and amine molecules eluted from the composite [5,6].

Previous studies have demonstrated the polymerization efficiency of TPO and BAPO [7–10]. These photoinitiators require a modification of the existing dental lights to extend the emission profile beyond blue light wavelengths for better curing. To accommodate that, several companies have developed new light-emitting-diode (LED) curing units that are capable of activating resin composites containing other photoinitiators, which highlights the commercial potential of these alternative systems. TPO and BAPO both have maximum absorption peaks ( $\lambda_{max}$ ) close to UV radiation, although their absorption profile extends toward the visible light region of the electromagnetic spectrum [8].

Despite the several benefits derived from photoreaction, the polymerization of composites is limited to the depth of light penetration. In composites, high filler loading causes light to be absorbed and scattered, and thus attenuated as it passes through the material. Polymerization is usually far more efficient on the surface close to the light source, and that efficiency decreases at deeper portions. This creates a gradient of polymerization within the material, and may result in undercured layers at the bottom areas [11]. It is known that the quantity, size, and shape of particles all affect the scattering of light through the composites [12]. A previous study reported that use of larger nanoparticles increased lightness

and reduced the yellowing effect of composites, whereas materials with smaller particles tended to have increased color stability [13]. However, to the best of our knowledge, no data is available about the relationship between alternative photoinitiator systems and particle size in nanostructured composites and their influence on optical properties.

The aim of this study was to establish a relationship between photoinitiator system and nanoparticle size on the optical properties and cure efficiency of model dental composites. The hypotheses of this study were: (i) composites formulated with CQ would present higher yellowing effect and higher color difference during storage than composites with BAPO or TPO; (ii) composites formulated with higher nanoparticle size would have lower yellowing effect and higher lightness than composites with lower nanoparticles; (iii) composites formulated with TPO and BAPO would present higher curing efficiency than composites formulated with CQ.

## 2. Materials and methods

### 2.1. Formulation of the model composites

Experimental composites were formulated by a 60:40 mol% mixture of BisGMA and TEGDMA (Esstech, Essington, PA, USA). Composites were prepared by incorporating 40 wt% of nanoparticles with either 7 nm (Aerosil® R 812, Evonik, Germany) or 16 nm (Aerosil® R 972) in average size. Fillers consist in hydrophobic fumed silica particles treated with hexamethyldisilazane (Aerosil® R 812) or dimethyldichlorosilane (Aerosil® R 972). Composites with three different photoinitiator systems added at 1 mol% were tested: CQ associated with ethyl 4-dimethylaminobenzoate (EDMAB), TPO, or BAPO, all from Sigma-Aldrich (Chemie, Steinheim, Germany). The photoinitiators were diluted in TEGDMA and then BisGMA was added. Fillers were gradually incorporated by hand-mixing and then the materials were mechanically mixed using a centrifugal mixer (SpeedMixer DAC150; FlackTek, Landrum, SC, USA) to produce a homogeneous paste. Six composites were formulated based on distinct filler size – photoinitiator system combinations.

Disk-shaped specimens were made in a circular steel mold (8 mm in diameter, 2 mm in thickness). After composite insertion, the top surface was covered with a Mylar strip and made flat by pressure with a glass slide. For each group, 12 specimens were obtained: six were used for optical properties analysis, and the other six for cure efficiency analysis. The specimens were light cured from the top surfaces for 40 s with a large spectrum LED curing unit (Bluephase G2; Ivoclar Vivadent, Schaan, Liechtenstein) with irradiance of 1200 mW/cm<sup>2</sup>. All specimens were mechanically polished in both surfaces in a grinding/polishing machine (Buehler, Lake Bluff, IL, USA) with a sequence of 2000- and 4000-grit SiC papers under continuous water cooling.

### 2.2. Optical properties analyses

The optical properties were determined according to the CIELAB parameters in the reflectance mode, with the specular component included (SCI) mode, over a zero calibrating box

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