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## Curing efficiency of dental resin composites formulated with camphorquinone or trimethylbenzoyl-diphenyl-phosphine oxide

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### ARTICLE INFO

#### Article history:

Received 3 August 2010

Received in revised form

1 November 2011

Accepted 10 November 2011

#### Keywords:

Camphorquinone

Degree of conversion

Photoinitiator

Resin composite

### ABSTRACT

**Objectives.** Since photoinitiator systems for dental resins based on camphorquinone (CQ) present color disadvantages, trimethylbenzoyl-diphenyl-phosphine oxide (TPO) has been proposed as an alternative. However, there are remaining considerations about its curing efficiency. The aims of the present investigation were: to characterize the relationship between the photoinitiator absorption spectra and the light spectrum emitted from a QTH light (absorbed power density,  $PD_{abs}$ ); to evaluate the kinetics of polymerization, and the depth of cure for filled dimethacrylate resins formulated with different photoinitiator systems.

**Methods.** CQ + EDMAB (control); TPO and TPO + EDMAB were used in 50:50 Bis-GMA/TEGDMA resins. Photoinitiator absorption and QTH-light emission were evaluated using a spectrophotometer and kinetics of polymerization with differential scanning calorimetry (DSC) ( $n=3$ ). Depth of cure was analyzed by the scraping method ( $n=5$ ), as recommended by ISO4049. One-way ANOVA/Tukey's ( $p<0.05$ ) was used to analyze the results.

**Results.** CQ presented higher  $PD_{abs}$  than TPO (364 and 223  $mW/cm^2$ , respectively). The DSC revealed that TPO and TPO + EDMAB produced a faster reaction than CQ + EDMAB. Composite formulated with CQ + EDMAB produced higher depth of cure ( $6.3 \pm 0.4$  mm) than those with TPO ( $4.3 \pm 0.1$ ) or TPO + EDMAB ( $4.2 \pm 0.3$ ).

**Significance.** Although CQ presented higher  $PD_{abs}$  than TPO, formulations containing TPO exhibited higher reactivity than that with CQ. On the other hand, materials formulated with TPO demonstrated lower depth of cure than that with CQ. Therefore, its use as an alternative photoinitiator requires further investigation, with higher concentrations.

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doi:10.1016/j.dental.2011.11.014

## 1. Introduction

The combination camphorquinone (CQ)/amine has been largely used as the photoinitiator/co-initiator system since the introduction of visible-light activated resin composites. One factor that influences radical formation in CQ/amine systems is the concentration of such components, which are known to vary among commercial brands [1]. There is evidence that higher concentrations of CQ improve the degree of conversion and mechanical properties of the formed polymer [2–6]. But above a certain threshold, no further benefits are observed [7] and in fact higher concentrations can affect esthetics due to the intense yellow color of CQ [4,6]. Since CQ is a bleaching type photoinitiator [8], its yellowing effect is reduced after the photoactivation process. However, part of the photoinitiator may remain unreacted due to insufficient irradiation [9] or other physical effects, such as the inner shielding effect (also known as “the inner filter effect”), where the light fails to reach the base of a given sample because the molecules near the surface absorbs most part of the light [7,10]. Therefore, color matching with the remaining tooth structure often poses a challenge [9,11].

Besides the “yellowing effect” that is commonly referred to in the literature, the combination CQ/amine system may present additional disadvantages. One such disadvantage is the potential toxicity of CQ [12]. The dependence of the co-initiator amine is another concern, due to potential toxicity of unreacted amines and to the fact that oxidative products may affect esthetics over time. Additionally, systems based on two components are extremely affected by the medium (resin) viscosity, imposing difficulties in mixtures like resin composites. Consequently, further research into simplified photoinitiator systems is encouraged.

Alternative photoinitiator systems that could be used in the formulation of dental resins have been proposed. One of the goals is to select colorless photosensitizers/initiators. The drawback with this approach is that these compounds are relatively insensitive to visible light. Stated in another way, their maximum absorption peak ( $\lambda_{\max}$ ) is located at shorter wavelengths than CQ [13], which raises a concern since most of the dental curing lights available on the market are focused at 470 nm, the  $\lambda_{\max}$  for CQ. One such alternative molecule is trimethylbenzoyl-diphenyl-phosphine oxide (TPO), a photoinitiator that presents absorption peak around 380 nm and is capable of functioning independent of an amine-type co-initiator. Therefore, TPO is a promising system for application in dental composites, especially considering esthetics, due to a potential reduction of the “yellowing effect” [14] caused by CQ and potentially higher color stability resulting from the absence of any amine components. On the other hand, few studies are available regarding its curing ability, especially for filled resin materials.

Therefore, the objectives of the present investigation were: to characterize the correlation between the light emission spectra from the curing unit with the absorbance spectrum of CQ and TPO – through calculations of the power density absorbed ( $PD_{\text{abs}}$ ); to evaluate the reaction kinetics of polymerization; and to determine the depth of cure of composites

formulated with CQ+amine, TPO, and TPO+amine. It was hypothesized that:

- i. CQ presents higher degree of conversion and a faster reaction than TPO when using wavelengths commonly emitted by traditional dental light curing units; and
- ii. composites formulated with TPO will produce lower depth of cure when compared with CQ, regardless of the presence of a co-initiator.

## 2. Materials and methods

### 2.1. Materials

The monomers 2,2-bis[4-(2-hydroxy-3-methacroyloxypropoxy)phenyl]propane (Bis-GMA, Esstech, Essington, PA, USA) and triethyleneglycol dimethacrylate (TEGDMA, Esstech) were mixed in equal parts by weight. Two photoinitiators were used to make the resin photo-curable: CQ (Polysciences Inc., Warrington, PA, USA) and TPO (BASF Chemical Company, Charlotte, NC, USA). A tertiary amine, ethyl 4-dimethylaminobenzoate (EDMAB, Avocado, Heysham, Lancashire, UK) was used as co-initiator. The photoinitiator/co-initiator systems tested are presented in Table 1. An inhibitor, 2,6-di-*tert*-butyl-4-methyl-phenol (BHT, Aldrich, Milwaukee, WI, USA), was added at 0.05 wt% to all materials. The concentrations were chosen based on a previous study on CQ/amine combinations where degrees of conversion were optimized at this level of initiator content [15]. We aimed at highlighting the point that, in spite of the higher absorptivity of TPO, its efficiency is much lower than CQ in terms of rate of polymerization when irradiated by a conventional, commonly used light source, because the irradiance is much lower at the wavelengths compatible with TPO. In other words, since we wanted to use a clinically relevant light source (and not adjust the emission spectrum to ensure equal irradiances at 380 and 470 nm), practical comparisons of the effects of the different absorption efficiencies were cleaner by using equal concentrations of initiators. Therefore, this study focused on the potential risks for conversion in depth, due to light penetration issues, with the use of alternative photoinitiators.

Part of the resins already mixed was separated and inorganic silanated fillers of strontium glass and fumed silica in a 15:1 ratio by weight were added at 80 wt%. All the components were mechanically mixed (DAC 150 Speed Mixer, Flacktek, Landrum, SC, USA) for 1 min to produce a homogeneous paste. All materials were prepared and handled under safe yellow light. All photoactivation procedures were carried out with a quartz-tungsten-halogen (QTH) light curing unit (LCU) (VIP, Bisco Inc., Schaumburg, IL, USA).

### 2.2. Methods

#### 2.2.1. Light curing unit emission and photoinitiators absorption spectra readings

The total light curing unit power output (mW) was measured with a power meter (Powermax 5200, Molectron, Portland, OR, USA). The irradiance ( $E$ ), in  $\text{mW}/\text{cm}^2$ , was determined by dividing the power output by the area of the light guide.

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