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## Degree of conversion and color stability of the light curing resin with new photoinitiator systems

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### ARTICLE INFO

#### Article history:

Received 22 April 2008

Received in revised form

10 March 2009

Accepted 10 March 2009

#### Keywords:

Degree of conversion

Color stability

Photoinitiator

OPPI

Light curing

### ABSTRACT

**Objectives.** This study investigated *p*-octyloxy-phenyl-phenyl iodonium hexafluoroantimonate (OPPI) as a photoinitiator, in combination with camphorquinone/amine photoinitiation systems, for use with di(meth)acrylate-based composite resins. The investigation determined if the inclusion of OPPI improved degree and rate of conversion, initial color and color stability of a representative composite resin dental material.

**Methods.** Camphorquinone (CQ) and OPPI were combined in various proportions with the amine co-initiator 2-dimethylaminoethyl methacrylate (DMAEMA) and used at two levels in which CQ + OPPI + DMAEMA = 1 wt.% or 3 wt.% to photoinitiate a BisGMA/BisEMA/TEGDMA (37.5:37.5:25 wt.%) monomer blend.

A total of eight groups (four groups for each level of total photoinitiator, 1% and 3%) were tested according to the following proportion of components in the photoinitiator system:

Group C: CQ only.

Group CO: CQ + OPPI (1:2).

Group CA: CQ + DMAEMA (1:2).

Group COA: CQ + OPPI + DMAEMA (1:1:1).

Each monomer was polymerized using a quartz-halogen curing unit (Demetron 400, Demetron Research Corp., Danbury, CT) with an intensity of 400 mW/cm<sup>2</sup> for 5 s, 20 s, 40 s, 60 s, 300 s and their conversion levels (DC) were determined at each exposure time using a Fourier transform infrared spectrophotometer (FTIR).

To examine color stability, experimental composite resins were made by mixing 3.2% silanated barium glass (78 wt.%, average filler size; 1 μm) with each monomer system, except both CQ only group and 1% CO group, which were found to cure insufficiently to be able to prepare useful specimens. Disk-shaped samples (10 mm in diameter and 1.5 mm in thickness) were made and stored under the conditions of dry or saline solution at room temperature (25 °C) or 60 °C water bath.

Each CIELAB scale was determined with a colorimeter (CHROMA METER CR-400) at the time of baseline (day after curing), 1 week, 2 weeks, and 4 weeks later.

**Results.** The high level (3%) photoinitiated groups exhibited greater DC than the low level (1%) groups. In the 3% group, the COA group showed the fastest and the highest DC, while in the 1% group the CA and COA groups showed the greatest DC.

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doi:10.1016/j.dental.2009.03.004

In the color stability test, both CA groups were darker and more yellow than the CO and COA groups. Color was more stable in composite resins containing OPPI than those containing only the CQ and amine components. The least color change (greatest color stability) was found using 25 °C saline solution aging, and the most change (least color stability) occurred using 60 °C dry air aging.

*Significance.* This study suggests that OPPI can be used to replace the amine in a given CQ/amine photoinitiator system to accelerate cure rate, increase conversion, reduce initial color and increase color stability.

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## 1. Introduction

Photopolymerization uses light energy to initiate photochemical and chemical reactions in organic oligomers, to form a new polymeric material by the photo-induced increase of molecular weight by monomer to polymer conversion, as well as crosslinking of developing or preexisting macromolecules. It has been noted that improved photopolymerization is critical for the optimization of mechanical properties [1,2], biocompatibility [3], and color stability [4] of light-activated dental resins.

Free-radical polymerization can be initiated by the excitation of suitable photoinitiating systems (PISs) under lights, since direct formation of reactive species on the monomer by light absorption is not an efficient way. Most of these systems were originally sensitive to UV lights, but by now a large number of various systems allow to extend the spectral sensitivity (that corresponds to the best matching between the emission spectrum of the light source and the absorption spectrum of the formulation) to visible lights [5]. In addition, the number and reactivity of these primary radicals modulate the early stages of polymerization kinetics, the polymeric chain lengths and even the final degree of conversion [6].

The visible light photosensitizer camphoroquinone (CQ), an alpha dicarbonyl that absorbs at 468 nm, is widely used in dental resin and adhesive formulations. This can produce a pair of free radicals by proton abstraction, besides this process can be efficiently accomplished by the formation of a complex between the photoexcited sensitizer and an electron donating (reducing) agent such as a tertiary amine [7]. CQ is a solid yellow compound with an unbleachable chromophore group, so that large amounts of CQ in resin formulations lead to an undesirable yellow color, affecting the final aesthetic appearance of the cured material [8–10]. This, in turn, places practical limits on the concentration of CQ and, consequently, limits the degree of polymerization and depth of cure that can be attained [11]. Therefore, there has been substantial effort to improve this curing system by the use of alternative photosensitizers [12–17] and various amine reducing agents [18,19]. In recent years, manufacturers have also included different photoinitiators in the organic matrix to act either alone or synergistically with CQ [20]. For example, the  $\alpha$ -diketone PPD (1-phenyl 1,2-propanedione) is used in adhesives and composite resin formulations to improve the polymerization kinetics and lessen photo-yellowing effects [11].

In this study, the onium compound *p*-octyloxy-phenyl-phenyl iodonium hexafluoroantimonate (OPPI) was used as a photoinitiator. It absorbs at 300–380 nm [21], which is outside

the range of visible wavelengths, and is therefore colorless.

Although it was reported that almost no polymerization occurred when OPPI was used alone [22], Eick et al. used this kind of OPPI as a cationic photoacid initiator in combination with a visible light sensitizer, CQ and an electron donor, ethyl 4-dimethylaminobenzoate (EDMAB) to polymerize an oxirane/polyol resin. In their study, photocuring was accomplished by the generation of a strong acid released during the generation of a strong acid released during the fragmentation of the diphenyliodonium salt photoinitiator. The acid protonates the oxirane groups and initiates cationic polymerization [23].

This study was carried out in order to determine if OPPI could be used as one component of a free-radical photoinitiator system, and can improve color stability in acrylic resins such as those used in dental (e.g., adhesives and restorations) and other biomedical applications. For this, OPPI was evaluated for its effect on the degree and rate of conversion and color stability of photoinitiated BisGMA-based resins.

## 2. Materials and methods

Monomer mixture (GTE) was made by mixing 37.5 wt.% BIS-GMA (lot # 568-21-07, ESSTECH, Essington, PA), 37.5 wt.% BISEMA (lot # 474-32-02, ESSCHEM Inc., Linwood, PA), and 25 wt.% TEGDMA (lot # 597-23-02, ESSTECH).

Total concentrations of 1 wt.% and 3 wt.% of variously proportioned photo-initiators (CQ/OPPI) or/and DMAEMA were added to make the monomer mixture curable. Eight groups (four groups in each concentration) according to the weight proportion of those components were tested (Table 1). Those kinds of works were done under filtered orange light.

### 2.1. Degree and rate of conversion

FTIR, HPLC, and NMR have mainly been used for the determination of the photopolymerization efficiency of dental resins. However, the use of FTIR absorption spectroscopy is the easiest and simplest method [16]. Therefore, degree of conversion (DC) was determined using a Fourier transform infrared (FTIR) spectrophotometer (Nicolet 520, Nicolet Instrument Corp., Madison, WI) in this study.

A small amount of uncured resin monomer (GTE) was placed between two NaCl disks and the spectrum recorded in transmission with 40 scans at a resolution of 1 cm<sup>-1</sup> (baseline). After the IR spectral scan, the monomer mixture was cured between the transparent NaCl disks for 5 s, 20 s, 40 s, 60 s, and

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