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# Effect of diphenyliodonium hexafluorophosphate on the physical and chemical properties of ethanolic solvated resins containing camphorquinone and 1-phenyl-1,2-propanedione sensitizers as initiators

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## ABSTRACT

**Objectives.** To evaluate the effects of the diphenyliodonium hexafluorophosphate (DPI) on the physical and mechanical properties of solvated dental adhesive resins containing camphorquinone (CQ) and/or 1-phenyl-1,2-propanedione (PPD) as initiators.

**Methods.** Model solvated resins containing bisphenol glycidyl methacrylate (BisGMA); triethyleneglycol dimethacrylate (TEGDMA); 1,3-glycerol dimethacrylate (GDMA); 2-hydroxyethyl methacrylate (HEMA); dimethylaminoethyl amine benzoate (EDAB) and ethanol were prepared. The resins were divided in 24 test groups according to the incorporated initiator systems (CQ-0.5 or 1 mol%; PPD-0.5 or 1 mol%; CQ + PPD-0.5 or 1 mol%) as well the presence of DPI (0, 0.5 or 1 mol%). Degree of conversion (using Fourier-transformed near infra-red spectroscopy), flexural strength and modulus by three point bending, cohesive strength and water sorption and solubility were measured. Data were statistically analyzed by one and two way ANOVA and Tukey's test ( $\alpha = 0.05$ ).

**Results.** DPI increased the degree of conversion of all materials tested. Camphorquinone promoted higher degree of conversion than resins containing only PPD or CQ + PPD. Generally, the resins containing PPD + CQ with DPI presented higher flexural strength and modulus, cohesive strength, as well lower water sorption and solubility.

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*Significance.* The use of PPD combined with CQ can increase the physical properties of the solvated resins. DPI improved the monomer conversion of all experimental materials and can positively modulate most of the physical properties of the solvated resins.

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## 1. Introduction

Camphorquinone (CQ) is the visible light absorbing photosensitizer component of the photoinitiator system most commonly used in dental resin materials. This sensitizer is a type II diketone, requiring a co-initiator, usually a tertiary amine, to producing free radicals and initiate the reaction [1]. Camphorquinone has an absorption range between 360 and 510 nm, with an absorption maximum peak at 468 nm [2] and is presented as a powder with intense yellow color [3]. High concentrations of this yellow initiator in resins can potentially limit usage in shades specially designed to restore bleached teeth.

Due to this issue, alternative photoinitiators have been investigated for either total or partial substitution of CQ, thereby reducing the amount of CQ in dental resins. The alpha diketone sensitizer: 1-phenyl-1,2-propanedione (PPD), can be used either alone (with a co-initiator) or in combination with CQ, to produce polymers less yellowing than the materials using only CQ [4]. However, unlike CQ, PPD has an absorption peak nearer to the UV region (393 nm) [5], requiring light curing units (LCUs) with a broad spectral output to promote reliable curing. An important characteristic of PPD is that this sensitizer can improve crosslinking by monomers in the network. The increased crosslinking improves the physicochemical properties of the resultant polymer [4,6,7].

Other agents that can improve the efficiency of light curing materials are co-initiators, such as diaryl iodonium salts. The diphenyliodonium hexafluorophosphate salt (DPI) is an initiator for UV-light curing systems and has ability to regenerate CQ molecules by replacing inactive and terminating radicals with phenyl active radicals and generating additional phenyl active radicals [8].

Improving photopolymerization efficiency is desirable; however, this improvement must be accompanied by superior chemical and physical properties. Several studies have demonstrated the efficiency of iodonium salts to improve the resin curing and properties [9–11]. But the effect of this co-initiator combined with PPD is still relatively unknown. Therefore, this study aims to evaluate the influence of CQ and PPD initiators, as well as different concentrations of DPI, on the physical and chemical properties of solvated dental adhesive resin systems.

## 2. Materials and methods

### 2.1. Adhesive preparations

A solvated base formulation was prepared by mixing the monomers bisphenol glycidyl methacrylate, 25 wt%

(BisGMA); triethyleneglycol dimethacrylate, 20 wt% (TEGDMA); 1,3-glycerol dimethacrylate, 10 wt% (GDMA); 2-hydroxyethyl methacrylate, 25 wt% (HEMA) – all of them donated by Esstech Inc., Essington, PA, USA – together with ethanol, 20 wt% (Merck KGaA, Darmstadt, Germany). The amine co-initiator: dimethylaminoethyl amine benzoate, 2 mol% (EDAB, Sigma-Aldrich Inc., St. Louis, MO, USA) was added to the base formulation even for the groups (Table 1) only using PPD as initiator. The use of an amine co-initiator with PPD followed many previous studies demonstrating improved polymerization efficiency of PPD combined with a co-initiator [7,12].

Twenty four experimental groups (Table 1) were created by varying the initiator systems (CQ and PPD) and their concentrations (0.5 or 1.0 mol%), combined or not with diphenyliodonium hexafluorophosphate (DPI, Sigma-Aldrich Inc., St. Louis, MO, USA) in 3 concentrations (0, 0.5 or 1.0 mol%).

### 2.2. Light-curing unit (LCU)

A polywave light emitting diode (LED VALO, Ultradent Products Inc; S. Jordan, UT, USA) with average tip irradiance 1400 mW/cm<sup>2</sup> was used for all light curing procedures. This LCU provides a spectral output covering both the 380–420 nm range (peaking at 400 nm) and 420–500 nm (with a peak at 460 nm) thus providing effective activation of both photoinitiator molecules (Fig. 1). The light energy supplied for all evaluations was 28 J/cm<sup>2</sup> (20 s activation).

### 2.3. Degree of conversion

Discs (4 mm diameter × 1 mm thick) were prepared using polyvinylsiloxanes molds supported between glass microscope slides. Measurements ( $n=5$ ) were performed using a Fourier Transform Near Infrared Spectroscopy (FT-NIRS - Vertex 70, Bruker Optics, Ettlingen, Germany). Monomer conversion was determined by evaluation of the pre- and post-curing, based on the peak at 6164 cm<sup>-1</sup>, corresponding to the absorbance of the vinyl-CH<sub>2</sub> [13].

### 2.4. Cohesive strength

To analyze the cohesive strength ( $n=10$ ) bar shaped samples (7 mm length, 1 mm wide and 1 mm thick) were prepared using silicone molds. The specimens were stored at 37 °C for 24 h. After that, they were held in a microtensile device using cyanoacrylate adhesive (Super Bonder, Loctite Ltda, São Paulo, Brazil), and then subjected to tensile forces in a universal testing machine (EMIC DL 2000) at a crosshead speed of 0.5 mm/min. The rupture area was measured using a digital caliper (Mitutoyo, Tokyo, Japan). The microtensile

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