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# Ultra-fast light-curing resin composite with increased conversion and reduced monomer elution

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## ARTICLE INFO

### Article history:

Received 24 November 2013

Received in revised form

13 February 2014

Accepted 25 February 2014

### Keywords:

Conversion

Kinetics

Elution

Irradiation protocols

Polymerization efficiency

## ABSTRACT

**Objectives.** To test the null hypotheses that photoactive resin composites containing a Type I photoinitiator would exhibit reduced DC or increased monomer elution at substantially short curing times compared with materials based on a Type 2 ketone/amine system.

**Methods.** Two experimental resin composites were prepared, using either Lucirin-TPO or camphorquinone/DMAEMA. Specimens were light-cured using appropriate spectral emission that coincided with the absorption properties of each initiator using different irradiation protocols (0.5, 1, 3, 9 s at 500, 1000 and 2000 mW/cm<sup>2</sup> for Lucirin-TPO based composites and 20 or 40 s at 1000 mW/cm<sup>2</sup> for Lucirin-TPO and camphorquinone-based composites). Degree of conversion (DC) was measured by Raman spectroscopy, propagating radical concentrations were collected by means of electron paramagnetic resonance (EPR) and monomer leaching was characterized using high-performance liquid chromatography (HPLC).

**Results.** The null hypotheses were rejected, except for a single irradiation protocol (0.5 s @ 500 mW/cm<sup>2</sup>). Lucirin-TPO-based composites could cure 20 times faster and release at least 4 times less monomers in comparison to camphorquinone-based composites. At 1000 mW/cm<sup>2</sup>, and 1 s irradiation time for curing times of 1 s, Lucirin-TPO based composites displayed 10% higher DC. The difference in polymerization efficiency of Lucirin-TPO compared with camphorquinone-based resin composites were explained using EPR; the former showing a significantly greater yield of radicals which varied logarithmically with radiant exposure.

**Significance.** Lucirin-TPO is substantially more efficient at absorbing and converting photon energy when using a curing-light with an appropriate spectral emission and otherwise a limitation noted in several previous publications. At concentrations of 0.0134 mol/L,

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Lucirin-TPO-based composites require a minimum light intensity of 1000 mW/cm<sup>2</sup> and an exposure time of 1 s to provide significantly improved DC and minimal elution compared with a conventional photoinitiator system. The use of a wide range of curing protocols in the current experiment has realized the significant potential of Lucirin-TPO and its impact for clinical applications, in replacement to materials using camphorquinone.

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## 1. Introduction

The degree of conversion (DC) of dental resin-based composites is a key physical property of the polymer since functional material characteristics, such as mechanical properties, volumetric shrinkage, wear resistance and monomer elution are significantly correlated [1]. Incomplete monomer conversion is a well-known drawback of dental resin composites, and the resulting monomer release [2] could at least partially account for failures observed clinically, including persistent post-operative sensitivity and/or the need for endodontic treatment, as well as secondary caries [3–6]. Further, the release of un-reacted monomers was suggested to be responsible for undesirable biological responses, such as cytotoxicity or disturbance of pulp progenitor cells differentiation even at non-toxic concentrations [7]. Therefore, incomplete polymer conversion and the increased potential for monomer release is likely to affect the delicate balance between healing and chronic inflammation in damaged pulp tissues [8,9]. Moreover, sub-optimal polymerization of resin composites may promote bacterial colonization at its surface [10]. It has also been suggested that release of degradation by-products from the monomers in dental resin composites can potentially influence biofilm formation and microbial survival at the material surface [11].

Clinically, the most common strategy to maximize DC and minimize monomer elution is to provide sufficient energy to the system by increasing curing time. Several works focusing on commercially available composites have emphasized the need to apply at least 20 s, but more likely 40 s irradiation to minimize the amount of eluted substances, even with the use of high irradiance LED lights [12–14]. As stated by Polydorou et al. [13], even 40 s irradiation “seems insufficient in order to prevent a high release of monomer”. Currently, the general consensus in the clinics to light-cure a 2 mm layer of CQ-based commercial composites is 20 s at about 1000 mW/cm<sup>2</sup> with commercial LED polywave lights [15,16]. The industry is challenged to combine improvement of DC and decrease of monomer elution with a curing time as short as possible to satisfy the demands of a time-conscious dental practitioner. One interesting approach to significantly reduce curing time (3 s) while reaching similar or improved DC is to replace the conventional camphorquinone/amine system (CQ) (Type II initiator) with a Norrish Type I monoacylphosphine oxide photoinitiator, namely Lucirin-TPO (TPO) [17]. In a subsequent paper by the same group, it was reported the increased reaction speed did not lead to any increase in shrinkage stress, cuspal deflection or marginal leakage, but significantly improved mechanical properties [18]. However, the impact of replacing a Type 2 with a Type 1 photoinitiator

with regards to monomer elution has not been investigated to date.

Hence, the aim of the present study was to test the null hypotheses that at very short curing times, TPO-based resin composites would not exhibit any difference with regard to (1) DC or (2) monomer elution compared to a conventional CQ-based experimental composite.

## 2. Materials and methods

### 2.1. Materials

Two experimental resin composites were prepared, based on a 70/30 wt% Bis-GMA/TegDMA resin (Sigma Aldrich), using two different photoinitiator systems in equimolar concentrations (0.0134 mol/L): either camphorquinone and dimethylaminoethyl methacrylate 0.2/0.8 wt% (Sigma Aldrich), or Lucirin-TPO 0.42 wt% (BASF), subsequently referred as CQ and TPO, respectively. CQ concentrations were chosen in order to be within the region of maximum DC according to Yoshida and Greener [19]. The corresponding equimolar concentration of TPO also falls within the range described as adequate with regards to DC, since Miletic and Santini reported that any concentration in excess of 0.43 wt% resulted in only very small increase in DC values [20]. Hence, the concentration of both photoinitiators can be considered to be in the optimal range, which enables the comparison of both initiating systems in optimal conditions.

The following fillers were introduced to the resin matrix: silanated barium glass (G018-186/K6,  $d_{50} = 3 \pm 1 \mu\text{m}$ , Schott AG, Germany) and methacrylsilane treated fumed silica (12 nm, Evonik Industries, Germany) at 65 and 10 wt%, respectively. This is summarized in Table 1.

### 2.2. Light curing

Specimens were polymerized in Teflon moulds of 2 mm thickness and 5 mm diameter, covered on each side by Mylar films, resting on a glass slide. For the EPR measurements, rectangular specimens of dimensions 5 × 2 × 2 mm were prepared, also in Teflon moulds. Light-curing was carried out with an AURA light device (Lumencor, USA) providing narrow spectral outputs. Irradiation could be applied independently at two wavelength ranges: 395–415 nm and 455–485 nm for TPO and CQ-based composites, respectively as shown in Fig. 1. Light irradiance calibrations were carried out directly at the tip of the optic fiber light guide (6 mm diameter) with good linear fit ( $R^2 > 0.98$ ), using a calibrated and NIST traceable deuterium tungsten light source (DH2000-CAL, Ocean Optics, Dunedin, USA). Calibration and spectral irradiance

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