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Effect of diphenyliodonium hexafluorophosphate on resin cements containing different concentrations of ethyl 4-(dimethylamino)benzoate and 2-(dimethylamino)ethyl methacrylate as co-initiators

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ABSTRACT

Objective. The present study evaluated the influence of diphenyliodonium hexafluorophosphate (DPI) combined with two different amines [ethyl 4-(dimethylamino)benzoate (EDAB) and 2-(dimethylamino)ethyl methacrylate (DMAEMA)] on the properties of model resin cements.

Methods. A comonomer base containing a 1:1 mass ratio of 2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane (bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) was obtained, after which 1 mol% of camphorquinone and 0.1 mol% of hydroxyl butyl toluene were added to the comonomer blend. Concentrations of co-initiators varied at 0, 0.5 or 1 mol% for DPI and in 1 or 2 mol% for amines (DMAEMA or EDAB). Silanated Ba–Al–Si glass (60 wt%) was added as filler. The combination of each amine and DPI concentration resulted in 12 formulations, which had the following properties analyzed: degree of conversion (DC), water sorption (W_{sp}) and solubility (W_{sl}), flexural strength (FS) and flexural

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modulus (E_f). Data for DC, FS and E_f were analyzed by two-way ANOVA and Tukey's test ($\alpha=0.05$) and W_{sp} and W_{sl} by Kruskal–Wallis and Dunn tests ($\alpha=0.05$).

Results. EDAB promoted a higher DC than did DMAEMA; however, DPI increased DC for all materials with DMAEMA. The physical properties of resin formulations containing EDAB were significantly better than those of groups with DMAEMA; however, DPI had a positive influence on the chemical and physical properties of the model resin cement containing DMAEMA, especially with higher concentrations of amine.

Significance. EDAB proved to be more reactive than DMAEMA, being less influenced by DPI. Resins containing a 1:2 CQ/amine ratio had better properties than those with 1:1.

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1. Introduction

Resin cements are low-viscosity composites that have been used to lute glass-ceramic restorations [1], and may be classified according to their initiator systems as self-cured, light-cured or dual-cured, combining both physical and chemical mechanisms [2]. Based on favorable properties such as low solubility, a wide variety of shades and good adhesion to dental and ceramic structures, these luting agents became the first choice for the cementation of metal-free restorations and pre-fabricated posts. Despite the fact that light-cured materials provide longer working time and show higher color stability [3,4] than dual or self-cure cements, their performance is impaired when the power of the light-source is attenuated [5], limiting the indications for these cements to clinical situations in which it is possible for the cements to be cured with high irradiance.

To improve the light-curing of resin-based materials, several methods have been evaluated, including: (i) the use of different photo-activation protocols [6–10], (ii) heating composite prior to the restorative procedure [11,12] and (iii) the use of different photoinitiator systems [13–15]. Diphenyliodonium hexafluorophosphate (DPI) is a salt that improves polymerization by forming higher quantities of free-radicals in conventional systems, such as those using camphorquinone (CQ), increasing the degree of conversion and positively influencing the properties of the polymer formed [14–16].

The reaction is improved due to the very low energy of the C–I bond present in the DPI molecule. After reacting with camphorquinone/amine, the C–I bond is irreversibly broken, providing two phenyl radicals that can further react with residual amines, abstracting a proton and creating a new free-radical [15,17]. In addition, DPI is capable of reacting with inactive CQ radicals formed during the polymerization process [12] and acting as an electron acceptor, decreasing the termination rate and enhancing initiation as new radicals are generated due to DPI fragmentation [17].

Resinous restorative materials usually have a binary initiator system. CQ, a widely used initiator in the light-curing of dental materials, cannot by itself generate high concentrations of free-radicals to promote suitable curing. Thus, amines are used as co-initiators to improve polymerization with this sensitizer [18]. In high concentrations, amines can increase composite susceptibility to oxidation, causing staining or discoloration of the restoration [19]. Conversely,

low concentrations may hamper the reaction with CQ, resulting in lower monomer conversion [13].

Ethyl 4-(dimethylamino)benzoate (EDAB) and 2-(dimethylamino)ethyl methacrylate (DMAEMA) are conventionally used in CQ-amine systems, and their structures are very different. EDAB is an aromatic amine capable of donating a H^+ , even at low concentrations [20,21], while DMAEMA has an aliphatic structure and a tendency to bond with O_2 , inducing formation of oligomers [22]. Due to these differences, evaluating the behavior of these compounds in a ternary initiator system containing DPI is important to determine which amine is the better choice for the system, as well as to identify a concentration suitable for improving the polymerization process as well as the properties of resin composites with CQ as an initiator.

Therefore, the aim of the present study was to evaluate the influence of amines (EDAB and DMAEMA) at two concentrations (1 mol% and 2 mol%), and the interaction of these agents with different concentrations of DPI (0, 0.5 and 1 mol%), on model resin cements containing CQ as the initiator. The properties evaluated were degree of conversion, flexural strength and modulus, water sorption and solubility.

2. Materials and methods

2.1. Preparation of the model cements

Experimental resin materials were prepared at a 1:1 mass ratio of 2.2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl]propane (Bis-GMA) and triethyleneglycol dimethacrylate (TEGDMA) (Esstech Inc., Essington, PA, USA). Camphorquinone (1 mol%) and butylated hydroxytoluene (BHT) (Esstech Inc.) (0.1 mol%) were added (BHT as inhibitor), blended and homogenized for 1 h at room temperature with a magnetic stirrer. The model blend was divided into 12 fractions, labeled RC1 to RC12, and groups were formed according to the concentrations of diphenyliodonium hexafluorophosphate (0, 0.5 and 1 mol% – DPI, Sigma–Aldrich, Milwaukee, WI, USA), ethyl 4-(dimethylamino)benzoate (1 and 2 mol% – EDAB, Sigma–Aldrich) and 2-(dimethylamino)ethyl methacrylate (1 and 2 mol% DMAEMA, Sigma–Aldrich) in the formulations. Each formulation was loaded with 60 wt% of 0.7- μ m average size silanated barium borosilicate glass fillers (Esstech Inc.). All chemicals were used without further purification.

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