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Effect of an acidic sodium salt on the polymerization behavior of self-adhesive resin cements formulated with different adhesive monomers

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ABSTRACT

Objective. To determine the influence of benzenesulfonic acid sodium salt 98% (BAS) and the activation mode on the polymerization behavior of experimental self-adhesive resin cements (SARCs) formulated with distinct self-etch methacrylates.

Materials and methods. Three catalyst-pastes using different self-etch methacrylates (2MP — bis 2-(methacryloyloxy) ethyl phosphate; 4META — 4-methacryloxyethyl trimellitic acid anhydride; and GDMAP — 1,3-glycerol dimethacrylate phosphate) and four base-pastes were formulated from the incorporation of different amounts of BAS associated to *N,N*-dihydroxyethyl-*p*-toluidine (DHPT). BAS/DHPT ratios were blended respectively based on the following final weight (wt%): control (0.0;2.0%), BP1 (0.5;1.5%), BP2 (0.8;1.2%) and BP3 (1.0;1.0%). Real-time polymerization kinetics (KP) was assessed for 10 min by FTIR spectroscopy. Maximum rate of polymerization (R_p^{\max}) was obtained from the first derivative of the curve conversion versus time. Degree of conversion (DC) was determined over 10 min and after 24 h. SARCs were tested in self and dual activation modes. Data of R_p^{\max} and DC 24 h were respectively analyzed by two-way ANOVA followed by Tukey's method ($\alpha=0.05$). **Results.** All factors and their interactions were statistically significant ($p < 0.01$). BAS caused an increase in DC in the first minutes of the KP analysis, especially in self-activated groups.

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After 24 h, all groups showed higher DC than those observed in the first 10 min. In general, the addition of BAS increased the Rp^{\max} . The GDMAP-based materials group showed the highest Rp^{\max} values when exposed to light but self-activated formulations with GDMAP or 2MP showed the lowest Rp^{\max} .

Significance. The addition of BAS increased the degree of conversion of SARC in the first minutes and 24 h after the polymerization reaction. This finding showed the importance of adding this type of salt during resin cement production to achieve better polymerization in the first minutes of the luting procedure specially when light exposure is not possible.

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1. Introduction

The simplified application of self-adhesive resin cements (SARCs) may decrease the technique sensitivity of luting procedures for dental prosthesis because, in theory, this type of material does not demand any dental surface treatment [1]. Additionally, the potential for chemical bonding between SARCs and dental substrates or acid-resistant ceramics (e.g., zirconia) has also contributed to the increased interest about these materials [2,3]. Since the release of the first commercial SARC in 2002, the dental industry has developed several similar products with varied compositions [4].

Current SARCs contain different types of self-etch methacrylates, which are primarily ionized by the water present in the material [1]. The most prevalent adhesive monomer is 10-MDP (10-methacryloyloxy-decyl-dihydrogen-phosphate) [5,6], which is able to demineralize and infiltrate dental tissues at the same time. Besides micromechanical bonding, these adhesive monomers can also develop chemical interactions with the calcium existing in the hydroxyapatite, or with metal oxides present at the intaglio surface of polycrystalline ceramic restorations [3,7,8]. Such bonding mechanisms have been attributed to the presence of chemical groups in the self-etch methacrylates, including carboxylic, phosphoric, or phosphinic groups [9–14].

Unfortunately, the presence of self-etch methacrylates in SARCs can also jeopardize the polymerization reaction. Previous studies have shown that acidic monomers may inactivate the tertiary amine (*N-N*-dihydroxyethyl-*p*-toluidine — DHPT), especially in self-cure cements [1,4,15]. The reaction between these two components forms a charge transfer complex that negatively affects the polymerization, decreasing the mechanical properties of the cement [4,11].

To avoid the interaction between the self-etch methacrylate and the tertiary amine, alternative activation systems and chemical components have been developed [1,4]. Some of these components are based on aromatic sulfinate amide derivatives, such as benzenesulfinic acid sodium salt (BAS). According to Ikemura et al. [14], these derivatives interact with self-etch methacrylates, preventing their reaction with the tertiary amine. It is suggested that this reaction not only releases the amine to react in the polymerization reaction but also generates free radicals to initiate the process, increasing the degree of C=C conversion (DC). Another study has shown that other types of aromatic sulfinate amide derivatives, such as sodium *p*-toluenesulfinate, can react with adhesive

monomers when added to experimental SARCs [16]. Although these studies showed interesting results in terms of polymerization behavior of the adhesive material, the effect of the salt concentration on the properties of SARC still needs to be determined.

Because of the problem of amine inactivation, SARCs may achieve very low degree of polymerization in certain clinical situations with significant light attenuation, such as the cementation of thick and/or opaque ceramic restorations or prosthetic posts [17,18]. Thus, the addition of aromatic sulfinate amide derivatives is also important to increase the DC in such critical scenarios. Therefore, the aim of this study was to determine the effect of two factors, namely, the accelerator/activator ratio (BAS/DHPT) and activation mode (self or dual cure) on the maximum rate of polymerization (Rp^{\max}) and DC of experimental SARCs formulated with different self-etch methacrylates. The hypothesis tested was that an increase in the accelerator/activator ratio and the use of a dual cure mode (as opposed to self-cure) would increase Rp^{\max} and DC.

2. Materials and methods

2.1. Reagents

The monomers urethane dimethacrylate (UDMA — Esstech Inc., Essington, PA, USA), bisphenol-A-glycidyl methacrylate (Bis-GMA — Evonik, Essen, Germany), triethylene glycol dimethacrylate (TEGDMA — Esstech Inc.), 2-hydroxyethyl methacrylate (HEMA — Sigma-Aldrich, St. Louis, MO, USA), bis 2-(methacryloyloxy) ethyl phosphate (2MP — Sigma-Aldrich), and 4-methacryloxyethyl trimellitic acid anhydride (4META — Esstech Inc.) were used as received. The phosphate monomer 1,3-glycerol dimethacrylate phosphate (GDMAP) was synthesized as previously described [19]. These acidic monomers are widely used in the commercial formulation of different adhesive materials in dentistry. Their bonding mechanism to the dental substrates could be interesting for SARCs formulations. Fig. 1 shows the molecular structure of the three self-etch methacrylates. Silanized barium borosilicate glass particles (2 μ m average size — Esstech Inc.) were used as fillers. The photosensitizer camphorquinone (CQ — Esstech Inc.) and the co-initiator ethyl 4-(dimethylamino) benzoate (EDAB — Sigma-Aldrich) were added to render the materials photosensitive. Benzoyl peroxide (BPO — Sigma-Aldrich) and *N-N*-dihydroxyethyl-*p*-toluidine (DHPT — Sigma-Aldrich) were also added to render a dual cure mechanism and deu-

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