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Probing the dual function of a novel tertiary amine compound in dentin adhesive formulations

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ABSTRACT

Objectives. A novel tertiary amine compound containing three methacrylate-urethane groups was synthesized for application in dentin adhesives. The synthesis, photopolymerization kinetics, and leaching were examined in an earlier study using this novel compound as the co-initiator (0.5 and 1.75 wt% based on the total resin mass). The objective of this work was to investigate the potential of TUMA (8-(2-(((2-(methacryloyloxy)ethyl)carbamoyl)oxy)propyl)-6,10-dimethyl-4,12-dioxo-5,11-dioxo-3,8,13-triazapentadecane-1,15-diyl bis(2-methylacrylate)) to serve simultaneously as a co-initiator and co-monomer (15–45 wt% based on the total resin mass) in dentin adhesive formulations. The polymerization kinetics, water sorption and dynamic mechanical properties of these novel formulations were determined.

Materials and method. The monomer system contained Bisphenol A glycerolate dimethacrylate (BisGMA), 2-hydroxyethylmethacrylate (HEMA) and TUMA (synthesized in our lab) at the mass ratio of 45/(55-x)/x. Two photoinitiator (PI) systems were compared. One initiator system contains three components: camphorquinone (CQ), diphenyliodonium hexafluorophosphate (DPIHP) and ethyl-4-(dimethylamino) benzoate (EDMAB) and the second initiator system contains CQ and DPIHP. The control adhesive formulations are: C0-3: HEMA/BisGMA 45/55 (w/w) and 3-component PI and C0-2: HEMA/BisGMA 45/55 (w/w) and 2-component PI. These controls were used as a comparison to the experimental adhesive resins (Ex-3 or Ex-2), in which x represents the weight percentage of synthesized co-monomer (TUMA) to replace part of BisGMA. The control and experimental adhesive formulations were photo-polymerized and compared with regard to the degree of conversion (DC), polymerization rate (Rp), water sorption and dynamic mechanical analysis (DMA) under both dry and wet conditions.

Results. C0-3 and Ex-3 formulations had similar DC, while the DC of Ex-2 formulation was higher than C0-2. The DC was similar when comparing the two- component with the

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three-component photoinitiator system when TUMA was used at the same concentration. DMA under dry conditions shows higher rubbery storage modulus for all experimental formulations, while storage modulus at rubbery region under wet conditions was decreased as compared with control (C0-3). There was no statistically significant difference for the DMA results under both dry and wet conditions when comparing two- and three-component initiator systems with the same TUMA concentration.

Significance. The newly synthesized TUMA could serve simultaneously as a co-monomer and co-initiator in the absence of commercial co-initiator. This study provides information for the future development of new co-monomer/co-initiator for dentin adhesives and dental composites.

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1. Introduction

Polymer-based composites have become the most common dental restorative material with a current use rate more than twice that of amalgam filling materials [1]. The durability of composite restorations does not, however, match that of dental amalgam [2–6]. The average clinical lifetime of posterior composite resin restorations is just 5.7 years due to secondary decay or fracture [7,8]. The dominant reason for failure of composite restorations is secondary decay and clinically, the failure occurs most often at the composite/tooth interface [9,10].

The composite is too viscous to bond directly to the tooth and thus, a low viscosity adhesive must be used to form a bond between the tooth and composite. The integrity of the adhesive and the adhesive/tooth bond is very important for the durability of resin-based dental restorations. The lack of durable and effective dentin adhesives is considered a major problem with the use of composites in restorative dentistry [9,10]. The monomers used in dentin adhesives are particularly critical and monomer selection exerts considerable influence on the properties, durability and behavior of dentin adhesives in the wet, oral environment. Much attention and effort has been devoted to the development of new monomers. These new monomers are one approach in the research community's quest to develop dentin adhesives that provide durable bonding at the composite/tooth interface [11–14].

Photopolymerization using a visible light source is a popular and convenient means of curing dentin adhesives and composites [15]. The majority of commercial methacrylate-based dental resins contain camphorquinone (CQ)/amine pairs as visible light photoinitiating systems. CQ is a light absorbing photosensitizer and the amine compound is an electron donor serving as co-initiator. Recently, a third component (often a diaryliodonium or sulfonium salt) is applied into the CQ/amine photoinitiating system to improve the visible light induced photopolymerization [7,16–20].

The co-initiator (amine compound) plays a critical role in the photoinitiation process [21]. The types of co-initiator as well as its ratio to the photosensitizer influence the quality of the polymerization. Many of the available amine co-initiators cannot be used in dental restorative materials because of cytotoxicity concerns [2,22]. The release of co-initiators has been

considered a source of adverse reactions [22], e.g. local and systemic toxicity, pulpal irritation, allergic and estrogenic effects.

A tertiary amine co-initiator (TUMA) containing three methacrylate-urethane groups was synthesized in our laboratory for application in dentin adhesives [23]. The results from an earlier study showed comparable degree of conversion with formulations containing 0.5% or 1.75% TUMA as compared to commercial amine co-initiators ethyl-4-(dimethylamino)benzoate (EDMAB) and 2-(dimethylamino)ethyl methacrylate (DMAEMA). Furthermore, there was no detectable leaching of the new co-initiator (TUMA), while 53.6% EDMAB and 11.2% DMAEMA were released. The results from this study suggest that TUMA is a promising co-initiator, which could prevent amine release from the final dentin adhesives.

The performance of adhesive formulations containing this new compound, (TUMA), as one of the major components (e.g., 15, 25, 35 and 45 wt%) was investigated in this study. The polymerization kinetics, water sorption and viscoelastic behavior of TUMA-containing formulations were compared with control formulations cured in the presence or absence of the amine co-initiator EDMAB. The purpose of this investigation was to determine the ability of TUMA to serve simultaneously as co-initiator and co-monomer in adhesive formulations. The hypothesis of this investigation is that there is no statistically significant difference in the viscoelastic properties of dentin adhesives with TUMA as the co-monomer when formulated in the presence or absence of EDMAB.

2. Experimental

2.1. Materials

Bisphenol A glycerolate dimethacrylate (BisGMA, Poly-science, Warrington, PA) and 2-hydroxyethylmethacrylate (HEMA, Acros Organics, NJ) were used as received without further purification, as monomers. TUMA (8-(2-(((2-(methacryloyloxy)ethyl)carbonyloxy)propyl)-6,10-dimethyl-4,12-dioxo-5,11-dioxo-3,8,13-triazapentadecane-1,15-diyl bis(2-methylacrylate))) was synthesized in our lab and used as the co-monomer with HEMA and BisGMA [23]. Based on our previous studies [12,15,24–27], camphorquinone (CQ), ethyl-4-(dimethylamino) benzoate (EDMAB) and diphenyliodonium hexafluorophosphate (DPIHP) were used as a three-component-photoinitiator

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