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Network structure of Bis-GMA- and UDMA-based resin systems

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ABSTRACT

Objectives. The commonly used dental base monomers 2,2-bis[*p*-(2'-hydroxy-3'-methacryloxypropoxy)phenylene]propane (Bis-GMA) and 1,6-bis(methacryloxy-2-ethoxycarbonylamino)-2,4,4-trimethylhexane (UDMA) require the use of a diluent monomer, such as triethylene glycol dimethacrylate (TEGDMA). The aim of this study was to measure double bond conversion of UDMA/TEGDMA and Bis-GMA/TEGDMA polymeric systems, determine the leachable portion, and analyze network formation by evaluating crosslinking and pendant double bonds.

Methods. UDMA or Bis-GMA was combined with TEGDMA in systematic increments and irradiated to form light cured polymers. Fourier transform infrared spectroscopy in the near-infrared region was used to measure double bond conversion. The leachable sol fraction was analyzed by ¹H NMR. Resin composites were formulated. Flexural strength was measured by three-point bending and volumetric shrinkage was determined with a mercury dilatometer. **Results.** The amount of base monomer greatly influenced double bond conversion, sol fraction, and crosslinking. Increasing base monomer concentration decreased double bond conversion, increased the leachable fraction, and decreased crosslinking and network formation. At mole fractions higher than 0.125, the UDMA polymers had significantly higher conversion than the Bis-GMA polymers. Bis-GMA polymers had higher leachable amounts of unreacted monomer, while UDMA mixtures had more crosslinking than the Bis-GMA mixtures. In regards to the physical properties of resin composites, increasing the base monomer improved flexural strength and decreased volumetric shrinkage.

Significance. This systematic study for the evaluation of conversion, leachability, crosslinking, and network structure along with physical properties, like volumetric shrinkage and flexural strength, are required for the optimization of competing desirable properties for the development of durable materials.

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1. Introduction

2,2-Bis[*p*-(2'-hydroxy-3'-methacryloxypropoxy)phenylene]propane (Bis-GMA) and 1,6-bis(methacryloxy-2-ethoxycarbonylamino)-2,4,4-trimethylhexane (UDMA) are commonly

used as dental base monomers in composite formulations, but their high viscosity and multiple functional groups require the use of a low viscosity diluent monomer, such as triethylene glycol dimethacrylate (TEGDMA). The structures of the monomers are shown in Fig. 1. When cured, these systems often have incomplete double bond conversion after

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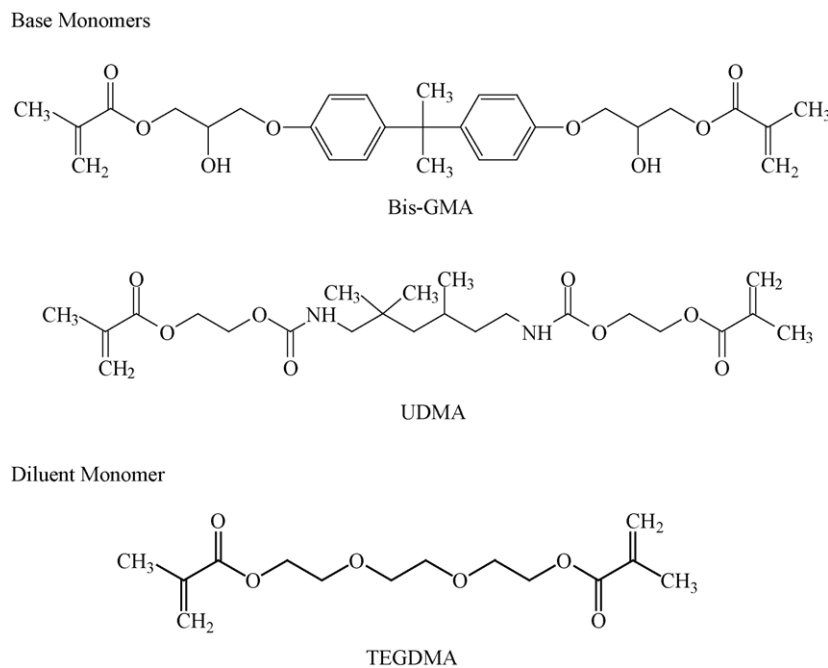


Fig. 1 – Structures of monomers used. For acronyms see Section 1.

polymerization [1–4]. As the polymerization progresses, the network formation decreases the mobility of monomers and oligomeric molecules resulting in pendant methacrylate groups and unreacted monomer trapped in the material. Low levels of conversion affect physical properties of the polymer, decreasing its strength [5].

It has been a common practice to measure the reduction in double bonds due to the polymerization process via infrared (IR) spectroscopy on thin films [6] and in bulk [7,8]. In crosslinking systems, residual double bonds in the polymer do not necessarily indicate free monomer, but may also result from pendant double bonds that are tied into the polymer network. Crosslinking is important for good network formation and physical properties. The extent of crosslinking, pendant double bonds, and leachable components have been analyzed by extraction of methyl methacrylate and crosslinking dimethacrylates from denture base polymers [9]. Also, crosslink density has been indirectly assessed by softening of polymers after exposure to ethanol [10,11].

If unreacted free monomer is left in the polymer matrix, the unbound components are able to diffuse out of the polymer system [12–17]. When this free monomer leaches out of the polymer, it may damage or irritate the surrounding tissue [18,19]. Pulpal cellular components can also be affected by the other components of the system, such as initiators or radicals [20]. The amount leached out of the polymer may vary with the type of monomers in the system and the degree of cure [21]. The toxicity of dental monomers also varies depending on the type and quantity of monomer [18,19,22,23]. Some monomers have been found to encourage bacteria growth [24]. Estrogenicity or allergic reactions have been reported [25,26], however, the estrogenic reaction may have come from impurities or degradation products.

To better understand the dependence of double bond conversion on monomer structure and reactivity of the monomer molecules in the forming network, these parameters were evaluated in a previous study [1] by photo-differential scanning calorimetry (DSC) using resin mixtures of TEGDMA with Bis-GMA or UDMA as base monomers. When compared at similar diluent concentrations, UDMA resins were significantly more reactive than Bis-GMA resins. Optimum reactivities in the UDMA resin system was obtained with the addition of relatively small amounts of TEGDMA, whereas the Bis-GMA/TEGDMA resin system required near-equivalent mole ratios for highest reactivity. Synergistic effects of base and diluent monomer on the polymerization rate and the final conversion were found for both Bis-GMA and UDMA base resins. The structures of the individual monomers, and consequently, the resin viscosities of the comonomer mixtures strongly influence both the rate and the extent of conversion of the photo-polymerization process.

This paper focuses on the network formation in bulk resin specimens of the same monomers, analyzing the leachable monomer fractions, and determining polymer crosslinking and pendant double bonds.

2. Materials and methods

The monomers Bis-GMA, TEGDMA, and UDMA (Esstech, Essington, PA), the photo- and cointiators camphorquinone (CQ) and ethyl-4-dimethylaminobenzoate (4-EDMAB), inhibitor 2,6-di-*tert*-butyl-4-methylphenol (BHT, Aldrich Chemical Co., Milwaukee, WI) and solvents dichloromethane and cyclohexane (J.T. Baker, Phillipsburg, NJ) were used as received.

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